THE JOURNAL

OF THE

AMERICAN LEATHER CHEMISTS ASSOCIATION

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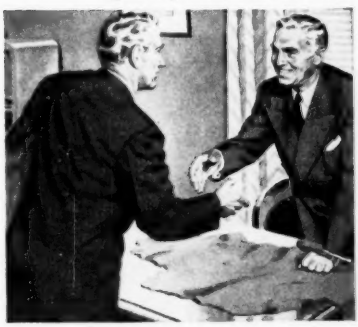

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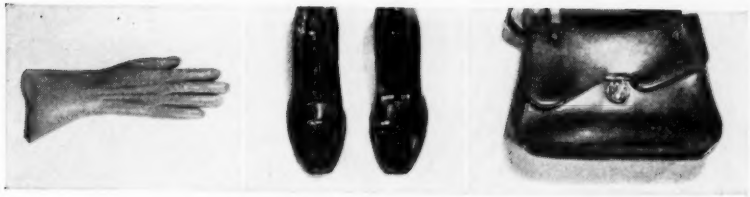

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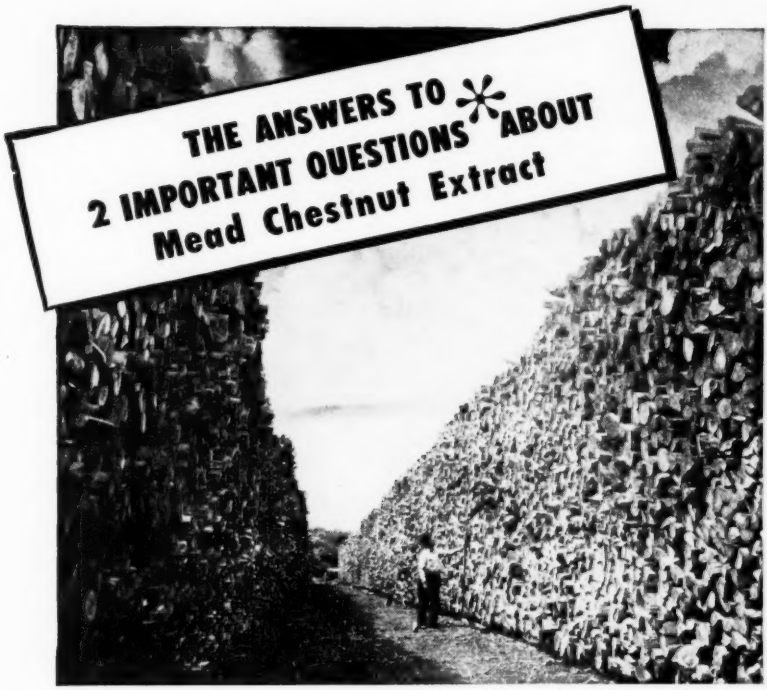

This is only a portion of the many stockpiles of chestnut logs that insure a continuous supply of Chestnut Extract.

1. Is the supply of Mead Chestnut Extract adequate?

Decidedly yes. Recent surveys show that there is enough chestnut wood in our forests to serve the leather industry for many years to come. Huge chestnut stockpiles, such as the one shown above, are converted under chemical control at five extract plants, insuring a steady supply of Mead Chestnut Extract.

2. Why is Mead Chestnut Extract preferred over other tanning agents?

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A. Chestnut tanned leathers do not oxidize, darken on exposure to light, or develop an uneven color on aging, as do leathers tanned with many other materials, vegetable or synthetic.

B. Since the tanning system demands acid, the use of chestnut, with its plumping acidity, saves you money.

C. Mead Chestnut Extract eliminates up to 28% of leachhouse losses.

D. There are no hidden costs. When you use Mead Chestnut Extract, no time- and money-consuming preparation is required.

Ask about Mead Cold Soluble Chestnut Extract for retanned leathers. And—for more pounds of plumper leather—standardize on Mead Chestnut Extract.

IT'S A FACT!

Mead Chestnut Extract will produce more pounds of plump leather per unit of tannin than any other tanning material.

Vegetable Tannin	Tan Yard Yield (*white weight basis*)
Chestnut Wood Extract	67%
Tanning Material A	61%
Tanning Material B	63%

Calculate your tanning material costs on a leather basis as well as on a tan unit basis. Mead Chestnut Extract, producing four to six pounds more leather, means positive savings.

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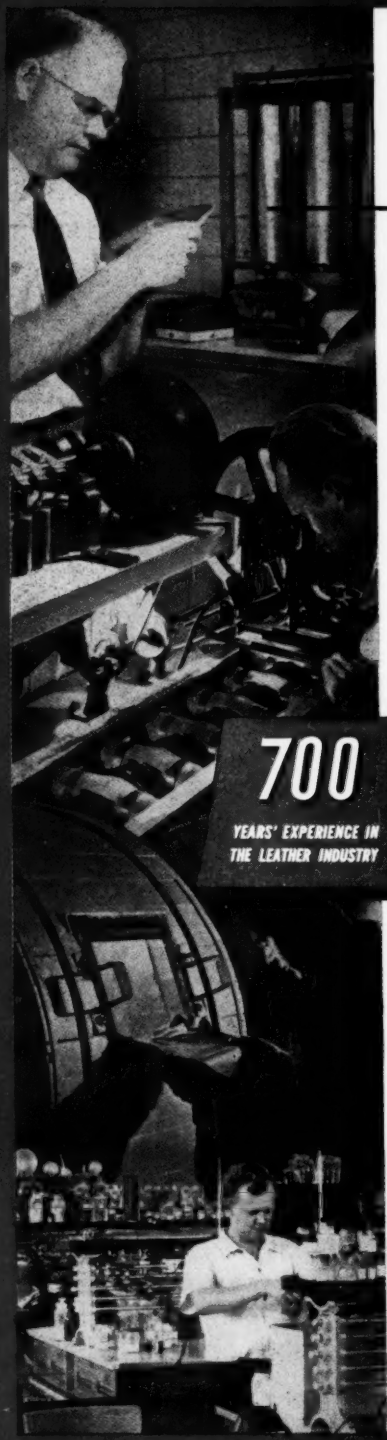

Leather Chemicals with a Valuable Plus

Leather chemicals were the products that started this company in business 44 years ago. Today the bates, synthetic tanning materials, and finishes we are supplying to our customers are the best we have ever made. Our standard is dependable, uniform quality.

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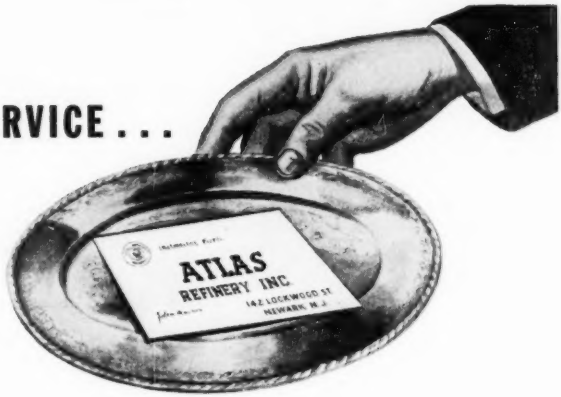

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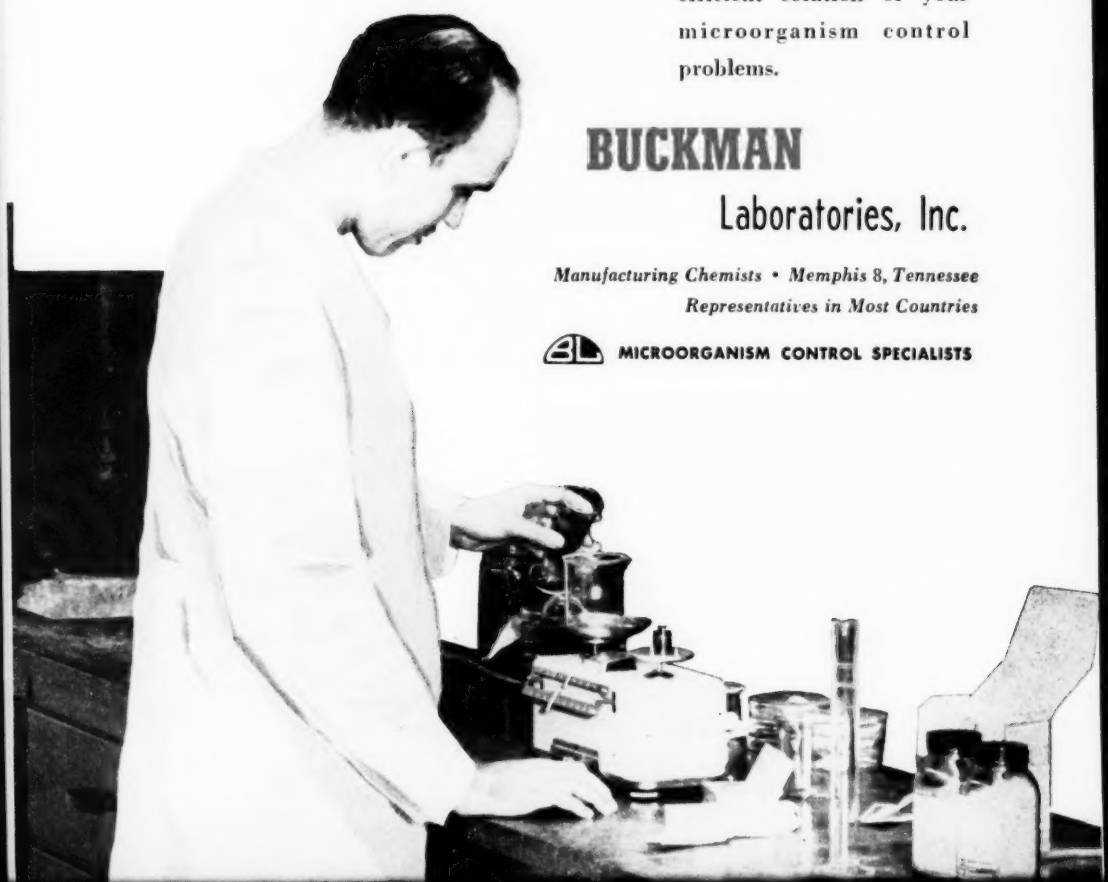

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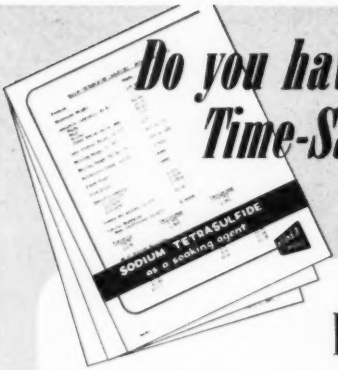

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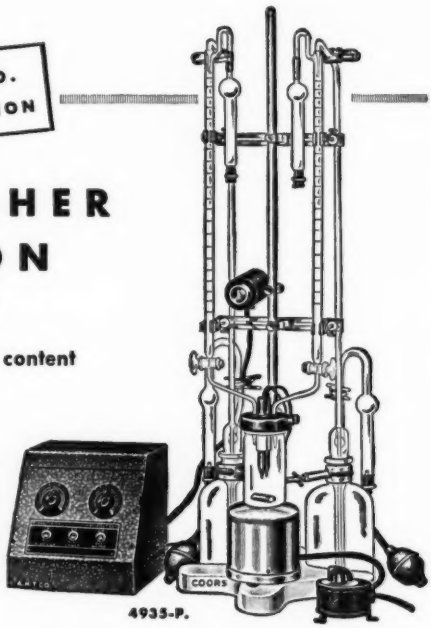

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4935-S. Ditto, 250 ml, with plastic cap . . . . . **.28**

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JOURNAL OF THE
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VOL. XLVIII OCTOBER 1953 No. 10

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POSITION OPEN: Sales Technician to call on South American Tanneries to demonstrate products and tanning methods. Must be willing to travel extensively. Fluent Spanish. Tannery experience essential. Salary open. Reply to the Diamond Alkali Inter-American Corporation, 122 East 42nd Street, New York 17, New York.

The American Leather Chemists Association†

By H. G. Turley, Ph.D.

Rohm & Haas Co.,
Philadelphia, Pa.

It is timely, on the occasion of the Fiftieth Anniversary, to present an account of the history and growth of the American Leather Chemists Association. We are proud that the ALCA, founded in 1903, is among the first ten of all scientific societies to be established in the United States, but if we do not include the national societies covering all aspects of chemistry and consider only societies exclusively devoted to the chemists of an industry, then the ALCA is second in age, exceeded only by the Association of Official Agricultural Chemists. It was through the efforts of a small group of Leather Chemists attached to the AOAC that the ALCA was born. Indeed, one of our early presidents characterized the Leather Chemists as a "caudal appendage" of the AOAC. In 1893 when the first attempt was made to standardize methods of Tannin determination, there were only six chemists employed in the leather industry which, at that time, was the third largest industry.

For details on the foundation of the ALCA, I am indebted to our first president fifty years ago, Mr. George Kerr, who is alive and well today and living in Virginia.*

Mr. Kerr writes, and I quote portions of his account, "The organization of the ALCA in 1903 was the outcome of a decade of sustained effort by a small group of young technological chemists to establish reliable analytical methods for determining the commercial value of all descriptions of materials employed in the manufacture of leather, and to introduce scientific methods of tannery plant control. As late as 1895 there was practically no recognized leather trades chemistry as such in this country; only a few tanners employed chemists in their plants and most of the analytical work called for was done by general analysis in the laboratories of those dealers in chemicals or dyestuffs who had taken on representations for tanning materials. It would be difficult for the members of the ALCA today to visualize the bitter disputes and the wrangling between chemists, to say nothing of the verbal abuse heaped upon the devoted heads of the pioneers of leather chemistry in the middle and late nineties and for many years later for that matter. Dr. Harvey W. Wiley was head of the Bureau of Chemistry at that time and evidently decided the subject of Tannin Analysis came within the orbit of the Bureau, inasmuch as he assigned one of his young assistants, W. H. Krug, to the task. The number of invitations sent out by Krug is unknown to me, but I remember distinctly there were only four in attendance including Krug. The most im-

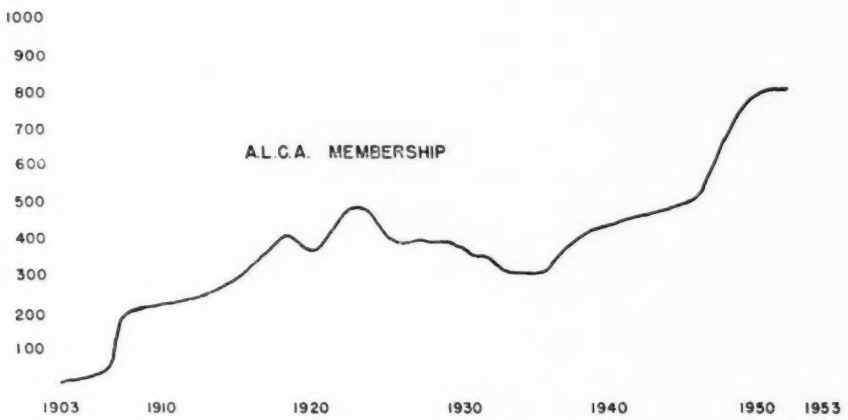
*We had hoped until very recently to have Mr. Kerr with us at this Meeting, but he has written to say that he will be unable to make the journey. I am sure you will all wish him good health and vigor, and on your behalf, we shall send him a telegram to that effect and add our congratulations and our respects to the founders of our Society.

†Presented at the Golden Jubilee Meeting of the A.L.C.A. in Cincinnati, Ohio, June 9, 1953.

portant action taken was the drafting of a provisonal method of Tannin Determination which would be suitable for commercial purposes. Following the meeting just described, each succeeding year saw its little gathering of the faithful and always there were a few more new faces, so while the number was so small it carried no weight with officialdom, the increasing interest of those attending the meetings gave reason to think that more rapid headway would ensue if an independent body could be organized, and at least two attempts were made prior to 1903 to do so. In due course came 1903 and the ALCA, the early days of which have been recorded more than once, sometimes with fair accuracy, sometimes not so accurately; however, the all-important point is not who did what or how, but the fact that the Association not only lives but has attained a strength far beyond the dreams of its founders and a value to the industry the magnitude of which is difficult to estimate."

The actual organization of the ALCA started with a small group of nine enthusiastic Leather Chemists who, having no official status in reporting analysis results as members of the AOAC, decided to form their own society in 1903. I would remind you that 1903 was also the founding of two great enterprises that have had phenomenal results on our human society. The huge aviation industry began in 1903 with the first successful flight of the Wright Brothers. 1903 also was the year of the foundation of the Ford Motor Company. Both these ventures prospered at a fantastic pace, with results that have changed the lives of every man, woman and child.

Now, how has our Association prospered over the span of fifty years? I have drawn up a curve of membership growth which includes Active and Associate members and not the Mutual members belonging to kindred European societies.



You will notice the very rapid growth of our Association which in about four years had two hundred members. Then note the steady growth to the period of World War I, and the slump coinciding with the sharp depression in 1920 just after the war. Then follows a gradual recession and a drop in members during the depression years of the early thirties. Then note again the gradual recovery to the outbreak of World War II. Some of my friends claim this result was governed by new political conceptions, but I prefer to disregard the fortunes of the Association as being influenced by politics. In the depression period the Association ran into financial difficulties from which we were firmly and, we trust, permanently rescued by the outstanding work done by a group of our members who unselfishly gave their time and energy to increase the income, mainly by increasing the revenue from advertising in the Journal. The Association will be forever indebted to these members and the strong solvent position of our Society today goes back to the splendid foundation laid down in the thirties. We now come to World War II and we see the sharp rise in membership, increasing by 60 per cent in the period 1945–50. It seems that the greater employment of chemists in the leather industry is a direct consequence of war—science expands tremendously, new fields of activity open up and even the traditional staple industries expand their chemical activity. You are all familiar with the government interest in leather in respect to specifications for supplies to the Armed Services and even to the support of research. Also, in the leather industry a number of fine, well-equipped laboratories were established by individual tanning firms. How astonished and proud the group of nine chemists in 1903 would be if they knew that today the ALCA has over eight hundred members.

Now, what of the future? I would be exceedingly rash were I to hazard a prediction as to the future of our Society. You will observe that in the past three years the growth curve has ceased to rise. The rate is approaching zero. If it becomes negative, then judging by the past we could predict disaster ahead. Fortunately, the old saying has been shown not to be true and "history does not repeat itself." As to whether we should strive to increase the membership is the concern of our Officers and it will be a question of whether we remain strictly the American Leather Chemists Association or whether we shall make concessions to embrace other excellent categories of those working in the leather industry. No doubt in the future we shall appeal to the membership —all of you—for advice on this point.

Turning to our Journal, the founders early realized the value of having their own independent scientific journal to record the deliberations of our Councils, to report the results of the work of our committees, to record information dealing with leather chemistry and leather making, and to point to the work of the rest of the world for the benefit of our members. Three years after our Association was founded, our first Journal appeared in 1906. This was a modest volume of 361 pages. The Journal has grown in prestige

and authority until today it is one of the world's outstanding journals devoted to the cause of leather chemistry. The Association has been especially fortunate in having able men of high principle as editors of the Journal, and these men have constantly and firmly guided the tone and style of the contents. I would mention our first editor, William Teas, from 1906–1910, then W. K. Alsop, editor for 26 years from 1911-1937, and finally, Dean Williams, editor for the past 16 years, i. e., from 1937 to the present time. The size of the Journal has grown over the years to the 1952 volume of 802 pages—the record being 1949 with 892 pages.

I call your attention particularly to the contents of our Journal over the years. All phases of leather making have been described and discussed, and much original research is found in the 47 published annual volumes. May I say that, in my experience, too many men employed in responsible positions in leather making are today still seeking simple information which I can assure them is to be found somewhere in the combined 29,873 pages of the Journal. My advice is that before seeking an answer to a simple question of leather technology or deciding that an idea is new, "First look it up in the Journal." The Journal is, of course, the medium for the reporting of the activities of our members, and I hope it will interest you to give a sketchy and all-too-brief summary of the nature of the work published by our members in the fifty years leading to our Golden Anniversary.

In the beginning, we were occupied almost entirely with perfecting the method of Tannin analysis, for indeed the original object of the Association was as stated in Mr. Teas' Presidential Address in 1906, "Solely the advancement and standardization of methods of analysis." Actually, we have never abandoned the search for an improved method for evaluating the commercial value of the world's tanning materials, and even today we have our Committee on Tannin Analysis under Chairman Arthur Kay still deliberating on the fine points of analytical technique in the endeavor to obtain results concordant among different laboratories. Mr. Kerr in 1905 stated the case for all time when he said, "It must be remembered that it (the method) is almost wholly empirical and like all empirical methods demands exactitude and attention to manual detail . . . " It seems that just as sure as we are of death and taxes, so too it is certain the Association will always be engaged on the problem of the Determination of Tannin. In those early days appeared many informative, descriptive articles dealing with some phase of leather making.

In the decade 1910-1920 changes began to appear because science was beginning to be used. In 1911 appeared a description of the method of measuring acidity by electr cal means, leading to the evolution of "pH"—a concept that has been of the greatest value to leather manufacturing and is commonplace today—yet there was, in the past, much argument as to the value of pH in the practice of making leather. Colloid chemists started making contributions, and a new school of chemical leather research was activated

by World War I. We now find Organic, Physical, and Bio- chemistry in addition to pure analysis. Enzymes began to be used and described, work on the different structures of the vegetable tans was in progress, and the concept of chromium complexes was initiated. Among the pioneers was J. A. Wilson who was among the first to point out that the chemist could be of greater value to the industry if his time and energies were turned to making better leather. In the decade 1920–1930 a real step forward was achieved by our chemists, and I mention the work of Wilson, Thomas, Gustavson and McLaughlin. In this period the important concept of measuring the acidity of leather was put forward by Kohn and Crede. This method of measurement using pH as an index of acidity shed a clear light on the complex and mysterious determination of acid in leather because it was realized that strong acid could be harmful to leather. This method won worldwide acceptance and is used today. Histology was used to study the architecture of skin and leather. In this period the Tanners' Council Laboratory was established at the University of Cincinnati, and there began that steady flow of research which continues today and which was recently published in six collected volumes, "Reports and Papers of The Research Laboratory of the Tanners' Council of America, Inc. at the University of Cincinnati 1924–1949." The names of McLaughlin, O'Flaherty, Highberger and Theis now appear frequently in the Journal. In 1927 a discovery was announced that was based directly on research, i. e., a new type of unhairing accelerator, the amines. Highberger commenced his basic studies of proteins and tanning which he continues today in the environment of a large research laboratory. Theis began his long series of publications on chrome chemistry which continued with patience and application until just two months ago. Two Presidential Addresses of that era appeal to me. President Smoot in 1924 hoped that the Association would drop too much work on the analysis of tannins, and Balderston in 1928 pleaded for the prosecution of true research for no monetary awards, adhering only to the spirit of science and the widening of human knowledge in our great field of leather chemistry. The contributions of the government chemists began to increase, and the classical work of Wallace, Bowker and Frey leading to the definition of safe leather adjudged by the lower limit of pH 3.0 has been of inestimable value to the leather industry and great monetary losses due to the deterioration or rotting of leather have been eliminated, and this principle is applied widely today.

In the fourth decade of our Journal, research continued at a higher level and some tanners established fine industrial laboratories devoted to tanning research. Yet in 1934 McLaughlin stated that the application of science had not kept pace with the now known scientific principles and, although some progress has been made, one might repeat McLaughlin's criticism today.

Now we come to World War II, the decade of 1940–1950, and leather again plays its part, although for the first time for the U. S. Army the traditional

sole leather was replaced by rubber. Research again jumps forward and new agencies are active in leather research, such as the Quartermaster Department of the Army's support of objective research on leather by the expenditure of public money. Specifications for leather increase greatly and analysis becomes of greater importance and increases in volume. This is the era of the application of physics to leather. New tools, such as the electron microscope, x-ray diffraction apparatus, spectrophotometry, and the polarograph were used to attempt to elucidate the structure of the parent protein, collagen, the mechanism of tanning and the constitution of chrome tanning solutions. Also, the physical testing of leather became correspondingly important and the Physical Testing Committee of the ALCA performed a great service in devising new methods, some of which evaluate unique properties, such as water resistance, air and water vapor permeability, and so on, and we pay tribute to Mr. M. Maeser–an engineer–and his committee members who still strive to solve the problems of leather testing. We must not neglect to mention the increasing contributions of micro-biology to the testing and proofing of leather against the ravages of insects and fungi in all kinds of severe climates. Tanning materials, domestic, vegetable, mineral and synthetic becomes of paramount value for the continuation of our supply of leather. The Eastern Regional Research Laboratory of the United States Department of Agriculture began its careful and sustained program of research on our domestic plant products bearing tannin. Many new products arrived permanently on the market. The classical and traditional iron tannage was improved and brought to a stand-by status to produce satisfactory leather for the public.

Throughout our existence some of our presidents have pleaded for the prosecution of research on a greater scale befitting our large industry and also to provide training schools for our young men planning to enter the leather industry. Perhaps compared with many new, vigorous, young industries established upon basic research, the leather industry has lagged behind and research has been done on too modest a scale. Nevertheless, a beginning has been made for training schools and the School of Tanning and Leather Technology has been re-established at the Pratt Institute which has already furnished about one hundred and thirty students for our own and foreign leather industries, and the State of Massachusetts has augmented the Lowell Textile Institute with a new School of Leather Engineering, well-endowed and splendidly equipped.

To encourage the interest of young men in our Association, your Council provided a new class of student membership at reduced fees so that the student could have the Journal at reduced cost.

In the past five years the Journal has continued to announce new research on new tannages, such as from aldehydes, resins, synthetic oils, in-situ tannages and new iron tannages.

In 1951, under President Koppenhoefer, the Association provided space on the program of the Annual Meeting for a Symposium conducted by practical men on practical, down-to-earth leather making problems, and the New England Tanners' Club had a successful meeting with discussions on soaking and liming. The Association will publish this Round Table Conference as a special supplement, and it is hoped that in the future other regional leather industry production clubs will continue these valuable discussions and that they will find a permanent record as supplements to our Journal.

In 1947 your Association joined the newly revived International Union of Leather Chemists Societies, and your delegates have attended the first meetings in Paris in 1949 and in London in 1951. It is hoped that delegates will be able to attend the third conference in Barcelona this year. These international meetings have begun in a fine spirit of cooperation and amicability, and the participation of our Society is especially welcomed by the group of Eurpean societies.

Let me repeat that we are all proud of the ALCA and its fine record, and on this jubilee celebration may we urge our young men to strive to play an active part and bring added lustre to the grand and ancient industry they have embarked upon. Join our technical committees, find renewed interest in your work, and strive to uphold and to improve leather in all its aspects of daily use, for we face new challenges today in a changing material world. Leather still maintains its place in the daily life of the population, although threatened, as in the past, with substitutes of amazing variability of performance and quality. Always remember that leather was the first "Miracle Fibre"—it provided man with protection and comfort in his environment in a savage world, and gave him long service and great utility coupled with beauty and an aesthetic appeal to the eye and to the touch.

In conclusion, at the Centenary celebration of our American Leather Chemists Association may it be said on that occasion that our founders and we builded well.

June 8, 1953

Sole Leather*

By L. M. WHITMORE

Leas & McVitty, Inc.,
Salem, Va.

It is unnecessary to tell this audience about the ancient art of tanning leather. *Some* people might think that remarks on sole leather should be entitled, like Longfellow's address to the Graduating Class at Bowdoin College on his Fiftieth Reunion "Morituri Salutamus", or "We who are about to die salute you." I am not that pessimistic.

*Presented at the Golden Jubilee Meeting of the A.L.C.A. in Cincinnati, Ohio, June 9, 1953.

The sole leather industry in this country was a gradual development from small tanneries in almost every community where the tanner was usually also a farmer or blacksmith, and the tan yard was really a yard and not a building. Time required in the usual process was from 18 to 24 months, and in the north during the winter the passing of time was about the only activity in these yards.

The next step was a gradual development of larger units which made a full time business of tanning, and were located primarily in relation to a good supply of Chestnut Oak or Hemlock Bark. Many tanneries now operating owe their location to these factors. This development is illustrated by some census figures:

> In 1894 the census showed 6,686 tanneries of all types, including one of Leas & McVitty at Shirleysburg, Pa. on land purchased from the sons of William Penn. Average workers per tannery 3.8—25,600 total.
>
> In 1899 the census showed 1,306 tanneries; some of which were local. Average workers per tannery 40—52,000 total.
>
> In 1939 census showed 446 plants, with an average of 106 workers per plant—47,000 total. 90 made sole or belting leather, and of these about 38 survive today.

CATTLE LEATHER TANNAGE IN U. S. — MILLIONS OF HIDES

Year	Total Cattle	Sole	Belting	Sole & Belting	% Belting & Sole
1899	15.8	7.7	.7	8.4	53
1929	10.1	7.2	1.0	8.2	43
1941	28.1	9.0	1.1	10.1	36
1951	20.9	5.2	.6	5.8	28
1952	21.0	5.2	.6	5.8	28

This statistical picture is not promising, but it must be remembered that the industry is not independent, but processes a by-product of the meat industry. It has been a hundred years since Buffalo were killed for their hides. It has been eight years since conditions have been such that any great number of hides could be imported, and the development of a large tanning industry in Argentina during World War II has eliminated the last large source of foreign hides, which prior to that time contributed from 4 to 8 million cattle hides to our supply. Prior to 1942, our own plants operated about 50 per cent on Frigorifices.

When speaking of sole leather tannage, I include belting, as many plants tan both sole and belting. However, the product of any so-called sole leather

tanner actually consists of about 50 per cent sole leather and the remainder shoulders, bellies and heads.

Heavy leather tanners have three problems that are peculiar to themselves.

(a) *Thickness.* The very nature of heavy leather intensifies the problems of penetration and proper distribution of tannage. Back in 1922 we started to analyze leather by strata to determine the extent and uniformity of tannage thruout the hide, as these data give information impossible to obtain by analysis of the entire sample.

The important factors in *Penetration and Tannage* may be briefly summarized:

Time. This was formerly 18 to 24 months, and fifty years ago was still usually 6 to 8 months, now 2 to 3 months.

Temperature. Originally depended entirely on the weather, later modified by the use of buildings, later heated buildings, and finally some semblance of temperature control. Years ago, I have seen packs frozen for a week with icicles on them.

Concentration of Tannin. Economy in use of tannin makes low concentration usual in early stages, but modern practice quickly builds up to 8 to 12 per cent by use of extracts. Previously the concentration was limited to the strengths obtainable in leaching.

pH. This concept was in my opinion the greatest single step forward in leather technology. It is not only important in actual tannage, but the final product we must admit is quite sensitive to pH conditions. Below 3 pH the fibres deteriorate, and above 5 the tannage tends to become unstable. The development of the glass electrode made accurate pH control practical.

Blend. The sole leather tanner making a product that carries a very narrow margin, now has to make use of a wide variety of natural tanning materials, and some synthetics of good quality in order to get a *blend that will give good results and not be prohibitive in cost.*
The heavy leather industry used to depend entirely on Hemlock and Chestnut Oak for tanning materials until about 1900 to 1910, when large supplies of Chestnut extract and Quebracho became available just as the supplies of the barks were becoming limited. Chestnut has a low natural pH and produces a very firm and high yielding leather.

Salt Content, Agitation of Liquor. Agitation of stock, size of tannin molecule, and concentration of non tannin are also important factors. Blends have to be formulated to give molecules small enough for good penetration, but too many of these will give low yield and low Degree of Tannage. The combinations are innumerable, but a satisfactory balance between penetration and fixation must be maintained.

(b) *Heavy Filling for Firmness.* Sole leather has to be well tanned and well filled for firness, and this brings in problems of drying and distribution of load unknown to the tanner of light leather.

(c) *Long Process Time* and consequently high investment per unit of production both in stock in proceess and in vat space. While a light leather tanner can buy his hides and sell leather from them in the same month, a heavy leather tanner must buy hides for three or six months before he can sell finished leather.

Wear of Sole Leather is often discussed with the comment that modern leather does not wear as well as the old long time tannages. Don't believe it. We took matched Bends and tanned by present process compared to the old 180 day process and used matched soles for testing. The results showed that the difference in wear was less than the limit of error in testing technique +2 per cent.

There is, however, a difference between the wear of leather made from the hide of a highly bred and pampered modern steer, and that made from the hide of an old time Texas steer which lived many years under rugged conditions. The fibre structure is different.

Wear testing technique is quite satisfactory and reproduceable results can be obtained if properly controlled tests are set up and about 10 matched pairs tested in actual wear. After doing a lot of work on abrasion resistance of leather we abandoned this entirely for practical purposes, as abrasion resistance, tho important, is only one factor in wear and final conclusions have to depend on practical tests anyway. With abrasion resistance varying 25 per cent from best to poorest areas of the Bend, and another 25 per cent from hide to hide, shotgun methods of sampling and testing are of little use.

The independent sole leather tanner operates in an intensely competitive area. He competes with plants operated by packers, who are his source of hides. He competes with tanneries operated by shoe manufacturers, who are his chief customers. He competes with upholstery and side upper tanners for hides. Last but not least he competes with synthetic soling materials which have greatly improved during the past few decades.

The status of synthetic materials is worth further analysis. The use of synthetics for soling has increased from practically nothing at the turn of the century to 59% in 1952. In 1918 there were at least 27 brands of synthetic soles, mostly rubber based. None survive to my knowledge. These were poor in quality, but gradually improved types have taken over a larger share of the market based on wear which was often much better than average leather. The recent development of the newer polymers such as synthetic rubbers and vinyl materials accelerated the improvement, so that current materials are a real threat to sole leather, especially high priced sole leather.

The newer synthetics, which have eliminated some of the worst features of the earlier products, are still inferior to leather in a number of points affecting not only foot comfort, but the actual protection of the foot:

1. They usually have low puncture resistance, especially when warm, less than 50 per cent that of leather.
2. Except for the air cell types, they usually have better thermal conductivity than leather, usually 20 to 50 per cent higher.
3. Protection of the foot from bruises and irregularities in the ground is often important, and a sole should be stiff enough to protect the foot but flexible enough to be comfortable. Leather meets this requirement in a high degree, while synthetics usually have a high thermal flexibility coefficient, and those that are satisfactory at higher temperatures are too stiff when cold, and *vice versa*.
4. Water vapor transmission is negligible in most synthetics, whereas leather is unique in being very effective in the transmission of water vapor, around 70 per cent as effective as free space. This ability to transmit water vapor is not a porous plate proposition. The leather simply absorbs moisture on one side and gives it off from the other if there is humidity differential to activate the operation.

This is especially important in keeping a normal low humidity in the shoe interior, with less deterioration of innersole and lining and less tendency to maintain fungus infections and various forms of dermatitis.

The above factors, in my opinion, have not been stressed enough in discussing the relative merits of leather and synthetics, and should outweigh the advantages the good synthetics undoubtedly have, cost and durability. Foot comfort is essential to normal activity, and should not be risked to save a few pennies on sole leather.

The development of synthetic soling material is, however, not entirely a calamity to the sole leather tanner. Most hides are being tanned, and if all shoes were made with leather soles, there would not be enough to go around. The availability of substitutes tends to stabilize sole leather prices, as any increase in leather prices automatically carries the threat of greater use of synthetics. Tanners would be well advised not to follow hides up beyond a point where they can make leather at prices competitive with substitute materials.

Sole leather was a natural target for synthetics and for many years sole leather tanners were the only ones much disturbed by substitute materials. Tanners of luggage, belting, upholstery and other special lines could outbid the sole leather tanner for native hides. Now that synthetics are being used in competition with leather for a wide variety of purposes, the sole leather tanner may get back onto a more equal footing in the competition for hides.

In conclusion, I feel that as long as cattle are raised, and beef is eaten, there will be hides to tan into sole leather, and many people will appreciate the value of comfortable hygienic shoes with leather soles. If so, the sole leather tanner will survive.

Received June 22, 1953.

A Half Century of Leather Coloring*

By R. H. LEACH

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The subject that I have been asked to discuss covers the coloring of leathers during the last half century.

It is not our intention to present specific formulae for the coloring of leathers as we have found generally that the exact shade produced is dependent on a great number of factors over which the dyer has little or no control. We are sure that everyone familiar with the coloring of leather recognizes this fact.

In assembling the data for preparation of this paper it was amazing to observe the similarity of some of the coloring processes of fifty years ago to today's methods. Let us go back to the early days and see what we had to work with and study a few of the processes generally applied.

Fifty years ago what type of leathers did we make? Among the usual types were black and colored calf, kid and sides, also, sheep, morocco and some splits, patent calf and sides. At the start of the present century we find that considerable progress had been made in the production of colored leathers. Also, the chrome tannages had passed the experimental stages so that in our discussion on the coloring of leather, both chrome and vegetable tannages must be considered.

Let us examine the dyestuffs we had in the early stages—excellent Phosphines were available, also Bismarck Brown and shading basic dyes, and the regular line of acid and direct dyes including the Resorcine Browns, Acid Oranges, Reds, Yellows, etc.

While the dyes used at that time were mostly imported, there were some dyestuffs being manufactured in this country.

Let us first study an early method for coloring black on chrome leather and compare it with a present procedure. Earlier it was common practice to use nigrosine, hematine and sometimes a striker of copperas, bichromate, etc. Direct black was also used in combination.

*Presented at the Golden Jubilee Meeting of the A.L.C.A., in Cincinnati, Ohio, June 9, 1953.

Today a mixture of direct black, direct blue, acid black and/or violet and hematine is used at the discretion of the tanner, and for extreme fullness, a slight topping of basic black.

Perhaps the greatest change would be in the manufacture of the direct blacks and the control of the uniformity of color, solubility and pH value. We also have the means of measuring the acidity or alkalinity today, whereas, years ago it was crude at its best, using litmus paper on the skins or relying on taste. We had no available method of controlling the acidity or alkalinity of dyestuffs either in their manufacture or solutions. Today it is possible to dye our leather in baths with controlled and reproducible acidity, measuring and controlling our pH with a glass electrode type of meter.

Thus we might say in coloring leathers in the fifty year period we have made changes that have improved our final results; first, for instance, improvement in the actual dyestuffs both in solubility and uniformity, secondly, our ability to reproduce uniform conditions in the leather prior and during the coloring operations, third, the development and production of dyestuffs better suited for the coloring of leather. Today we do not have to use dyes that are subject to color changes with a slight variation of acidity because dyestuffs have been produced especially for leather that are quite uniform in color over the range of pH that one finds in normal chrome leathers. Today we have also a choice of dyestuffs relatively fast to light when applied to chrome leather.

It is also possible for us to select dyestuffs to meet certain specific requirements such as dyestuffs as a group for penetration. We can also select dyestuffs which are definitely surface dyeing colors only but have good qualities due to the fact that they are level dyeing and the resulting shades are clear-toned.

From the wide selection of dyestuffs that are available today we can choose a homogenous dye that is quite close to our desired shade and eliminate the necessity of using shading dyes which are radically different in tone.

With the introduction of pigment finishes prior to World War I a greater field for colored leathers was opened due to the fact that the method of applying these finishes covered the leather to such an extent that the tanner was able to take a relatively poorly dyed skin and make a saleable piece of colored leather. This was indeed fortunate at that time on account of the extreme shortage of dyestuffs for it enabled the tanner to greatly curtail the percentage of dye used and meet his demands for finished colored leather.

On account of the shortage of dyestuffs during the war period, it was only natural for the tanner to turn again to the use of dyewoods with which he had been familiar over a long period of years. The principal ones being hypernic, fustic and hematine, which were used in conjunction with sumac, gambier, chestnut, etc. With the use of the proper strikers such as copperas, bluestone, sodium or potassium bichromate and potassium titanium

oxalate, it was possible to produce shades from light tans and browns to a deep mahogany and black.

As an illustration, a very popular shade at that time was mahogany and this was produced with a combination of hypernic—fustic—hematine struck with bichromate and topped with a small amount of Bismarck Brown.

This method was more or less discarded on account of the harshness, woody feel and coarser break imparted to the leathers. After the war when dyestuffs became more plentiful it was only natural that the tanners reverted to the usual method of mordanting with a small percentage of extracts and dyeing full shades with aniline colors which produced better leather in depth of shade and also gave superior results in both feel and break.

In the earlier part of the century considerable so-called "buck" leathers were made both in colors and white. This type of leather was made from kip or larger sides and was pearled and crusted like suede calf. When properly conditioned for cuffing it was buffed on the grain side instead of the flesh and a very short, close nap was produced.

A general coloring method for this leather was to wet back thoroughly, wash and then use a combination of gambier, sumac or chestnut, fustic, hematine in conjuction with acid or acid and chrome dyestuffs, strike with copperas or bichromates, depending on the shade,—wash, and then top with a combination of bolted white, blue clay or ground slate and earth colors such as the Siennas, Umbers, Red Oxides, etc. This bath of pigments was added through the door dry or was previously ground in a moist condition in a paint grinder, run in a short bath and then fat-liqoured with a small amount of sulfonated castor oil and egg yolk.

The finishing of this leather was done either by buffing on a Buzzell machine or blocking with a fine emery on a staking machine, after which it was brushed and then dusted on the table with a dry mixture of the same pigments, care being made to see that the dusting powder matched the shade desired.

Some of the heavier shades were topped with a small amount of basic dyes and the subsequent dusting with powder was eliminated.

Although buck leathers were made for a great many years, nobody successfully produced and put into production a black. Many attempts were made but due to the shortness of the nap it was almost impossible to add sufficient fatliquor to develop the color without matting or causing a sheen when handled.

Due to considerable objection by the general public to the extreme crocking of the colored buck produced by this method of coloring and finishing, it gradually lost favor around 1924–25 and for some years, aside from white, very little was produced.

Later this type of leather was colored from the shaved condition, thoroughly penetrated and then buffed on the grain side to produce the shades desired.

This presented a difficult coloring problem because it called for penetrating the leather at a sufficiently low pH so as not to have loose or coarse breaking leather when finished. The better penetrating dyestuffs were selected from the acid and chrome groups and these seemed to give the best results and this method of manufacture and coloring is still used at the present time.

It will be noted that this method of coloring has overcome to a great extent the extreme crocking which prevailed in the earlier method. With this method we are not depending on loosely held pigment powder to obtain our shades, but rather on the actual shade of the dyed leather fibres. Whatever dust is produced with this latter method may be removed to an acceptable point by the usual brushing machines.

It would seem that as long as we are discussing leathers which are buffed to produce their finished surface that we should now consider suede leathers.

Originally sueded leathers were made from small skins which were unsuited to finish for grain leathers, either due to imperfections on the grain or lack of tensile strength of the grain. We have in mind particularly skins from unborn calves which we know as slunks, the grain of which, in a great many cases, is so tender that ordinary machine operations of unhairing or fleshing tend to crack or break the grain surface. These defects on the grain surface have no appreciable effect on the flesh side and therefore it was only natural that a process was developed to finish these skins on the flesh side and produce what was termed sueded leathers.

The method of preparing the skins from the chrome tanned and shaved condition has changed very little with the years and our preparation or pearling, as we term it, with very few changes, is the same today as it was at the start of the present century.

This method of pearling can be considered to be as follows; after proper washing and neutralizing the skins were treated in a bath of egg yolk or substitute together with fillers such as flour, clays, gums, etc. This stock, after drying and proper conditioning with moisture, was staked and then buffed on an overshot wheel—first with a relatively coarse emery and then finished with finer emery. In this way the sueded nap was produced on the flesh side of the skin and could be considered in proper condition to be wet back and colored. Some suedes are still produced in this manner. The improvements that have been made in the past fifteen to twenty years have been due mainly to the development of buffing machines which can be so skillfully adjusted that a more even, closer nap can be produced over a larger area of any given skin.

An early method of coloring black suede calf which was used for quite a number of years was a combination of hematine-fustic-Nigrosine struck with copperas, washed, topped with basic black and fat-liquored with a sulphonated cod-oil. Then the stock was dried out, crusted for a few days, dampened in moist sawdust, staked either by knee or machine, dry milled

for a period of hours and then tacked on boards to dry. We note that this method of coloring seems to be an extension of the previous method given for grain leathers, although in the case of suedes greater quantities of dyes were necessary in order to give the desired fullness.

It was found that suede colored in this manner gradually became weaker so that if held over from one season to another it would not stand lasting and stitching. This deterioration in strength is something that a colorer of leather frequently encounters and is undoubtedly due to the penetrating dyes, dyewoods and strikers which have overtanned the leather and made the fibres more brittle. Experience has taught us that there are today many dyes which will penetrate the leather and have little or no detrimental effect on the strength.

While the previous process was in use experiments were being carried on with an entirely different method of dyeing black suede, namely that of diazotizing and developing. Leather colored with this method was actually in production as early as 1910. It was possible to produce leather either penetrated or with a white cut at the option of the tanner. Some preferred the leather with the white cut because it proved to be a much stronger type.

This process continued essentially the same until the early 1930's. At this time a radical mechanical change in procedure was made. We refer to the process known as top buffing both on blacks and colors. With the introduction of this extra color buffing, it became necessary to color and penetrate the skins to a sufficient depth so that particularly in the case of blacks we would have a good body of color even after top buffing. In addition to suede leathers made with the diazo type of dyestuffs we also used selected dyes from the acid, direct and basic groups.

A general method for the diazotized and developed coloring would be the application of the dye bath at 120°–130°F. penetrated sufficiently and exhausted with either formic or muriatic acids, cooled to 70°F. by washing and then diazotized by the introduction of sodium nitrite and muriatic acid and run thirty minutes. It was then washed with water at 70°F. for approximately thirty minutes to eliminate any excess of nitrous acid. Then, in a fresh bath of 70°F. the MTD developer was added and run 30 minutes, after which the stock was washed and warmed for the fat-liquor bath, either with or without a topping dye.

In the past number of years a further development in this process has been the use of sulfamic acid before the developing bath. This product is introduced after the diazotizing and eliminates the necessity of washing prior to developing and thus shortens the running time and also prevents any excess of nitrous acid from reacting with the developer.

Suede kid which had been produced in limited quantities prior to 1930 came into great prominence with the introduction of the top buffing method. We have found that the methods of coloring suede calf and suede kid are

quite similar with one exception, generally it is much more difficult to penetrate the grain layer of the kidskin. This difficulty has been overcome with the use of detergents or wetting back agents and by increasing the percentage of alkali in the wetting back and coloring operations, Here again we make our selection of dyes from the acid, chrome, direct and basic groups and in some cases we use in combination both penetrating and surface dyestuffs.

Experience has taught us that with a higher pH value we can penetrate ordinary durface dyeing colors sufficiently for top buffing, and in the same bath with the introduction of the proper dyes selected from the penetrating groups obtain the desired penetration of the grain layer.

Kid suede has for a great many years been produced in considerable quantities in shades ranging from delicate hues to the full, rich tones of browns, blues, greens and reds and plays a very prominent part in today's manufacture of sueded leathers.

One class of leather which has been developed to the greatest extent in recent years is chrome split leathers for suede and non-slip linings for both women's and men's shoes in the medium and lower price ranges.

The early production of split leathers was mainly gussets, work shoe uppers and yellow glove. Splits that were used earlier in the production of gussets and yellow glove apparently were not sorted for selection, while today only the poorer types would be used for this purpose. Today a great deal of care is taken in the sorting of splits, the better grades being used for the manufacture of suedes and non-slip lining leathers.

From our experience through the years in the manufacturing of suede calf and kid it was only natural that such knowledge should be applied to split leather. Since these splits may be received by the tanner from various sources and various tannages, it is first necessary to bring them to as uniform a tannage as possible.

After the splits have been split and shaved to the desired weight they are then conditioned for the chrome retan which may consist of 5 to 10 per cent of a prepared one bath chrome tan, and possibly one or two per cent of sodium formate. These splits may be neutralized to a pH value of four to five at the option of the tanner. They are then washed and fat-liquored in a manner somewhat similar to suede calf and kid, although due to the coarser structure of the fibres, more oil is generally required.

Several methods are used in the drying, conditioning and buffing of splits prior to coloring. It is most important that care be taken in the buffing so as to eliminate the possibility of burned spots or the nap being cut too close in the butt and along the backbone on account of sheen appearing after stock has been top buffed after color.

The preparation for the coloring of splits is similar to calf and kid suede and we also make the selection of dyestuffs from the same groups, namely the acid, direct, chrome and basic. The coloring of this class of leather, par-

ticularly the heavier shades, calls for a considerable percentage of dyestuffs and such shades produced without the use of basic dyes seem to give the best results, both in uniformity and clearness of tone, when finished buffed.

In the coloring of black suede splits we have found generally that the use of basic dyes in the coloring formula seem to aid in giving greater depth to the black. Black is also produced in some plants by the use of the diazotized and developed method.

It is also possible to help both nap and color in a black with the use of a singeing machine. A gas flame is used in such a machine with the flame being above the exposed surface of the split which is on a metal bolster moving at a regular speed. This flame is adjusted so as to burn the longer fibers and produce a closer nap. Occasionally, dark shades of Brown and Navy Blue are treated in this manner and if proper care is exercised excellent results are obtained.

The production of the splits for linings or non-slips is carried on in one operation from the shaved condition. After coloring and fat-liquoring the stock is dried out either by wet toggling or tacking and is finished by buffing after a staking, if necessary. Generally the shades produced range from grays, beiges, etc. and the regular line of acid dyestuffs is used in conjunction with various extracts, pigments and neutral syntans.

Since we have mentioned the use of syntans in the coloring of the lining splits it would be logical to discuss them at this point. Although the original German patent of Dr. Stiasny was granted in 1911, the use of both acid and neutral syntans did not become general in this country until possibly ten or twelve years later.

What properties are imparted to the leather by the use of syntans: First—we notice when syntans have been applied to chrome tanned stock that a considerable bleaching has occurred. Utilizing this effect, and in conjunction with white pigments, it is possible to produce satisfactory white tannages.

Second—we find that when acid and direct dyestuffs are applied to the syntan treated chrome leathers that the shades obtained are much lighter and increased penetration of color is apparent. At the same time we notice that there is a greater uniformity of shade over the entire area of the skin. Thus it is now possible with small percentages of dyestuffs in conjunction with syntans to produce the whole range of pastel shades.

Third—when chrome tanned stock is treated with syntans we find that an excellent mordant has been produced for the application of basic dyestuffs. Leather mordanted in such a manner exhausts basic dyestuffs to a greater extent and increases brilliance and clarity. We realize that syntans have a great effect on the solubility of extracts and have considerable effect on extract tannages. We feel that it is beyond the scope of this paper to discuss syntans other than their use in connection with dyeing.

During the past half century considerable progress has been made in the development of chrome tanned sheepskins, particularly for garment and

millinery types of leathers. Earlier coloring of sheep was done mainly on vegetable tannages with a relatively small percentage tanned in chrome. Colored linings were produced for the shoe trade, skivers for bookbinding and there also was a fair amount of suede. Blacks and browns predominated with a certain amount of natural extract colors and also alum whites.

Today there is a considerable demand for a leather which must be mellow, soft with a silky feel, finished either on the grain or flesh side as desired and has excellent draping qualities. It is apparent that the very nature of a sheepskin has all these properties, but we must also consider one more factor and that is eye appeal in order to make the leather satisfactory for millinery and garment purposes.

Each spring and fall certain shades are selected which must be matched closely on both leather and fabrics. These shades cover an extreme range from delicate pastel tints to the full, rich tones of browns, greens, reds, etc. As previously stated, we produce these pastel shades with the properly selected dyestuffs with the assistance of the syntans. Years ago with the demand for relatively fewer colors it was quite within the ability of the tanner to produce such shades as needed. Today, however, with the demand for such an extreme range of shades on all kinds of leathers it is necessary for the dyestuffs concerns to employ a considerable number of experienced technicians. These men with their knowledge of dyestuffs in cooperation with the tanners can submit approximate formulae for any desired shade.

In this review of the changes that have occurred in coloring leather during the first fifty years of the American Leather Chemists' Association, we have touched only the highlights. From the elementary blacks and browns of 1900 we have developed the great number of colors that are available to the consuming public today. This has not been forced on the tanner from an outside source but rather it has been his desire to create new and different leathers. We cannot predict what will be said to the American Leather Chemists' Association on the occasion of its 100th anniversary, but we believe that again continued progress toward more durable and beautiful colors will be reported.

The author wishes to express his appreciation for the assistance given him in the preparation of this paper by Mr. E. D. Lord of J. S. Barnet & Sons, Inc., Lynn, Massachusetts.

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"Development of Tannery Machinery in the United States During the Past Fifty Years"*

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Most of the facts and dates which I am going to mention have been gathered from various books and publications, and do not represent firsthand knowledge on my part. In other words, I personally have not spent the past fifty years in the tanning machinery business. The reason I was asked to give this paper is because the company which I represent has had an interest in the business since 1920 as a result of owning stock in the Turner Tanning Machinery Company.

Six years ago, my company embarked on a Research and Development program aimed at the modernization of tanning machinery. Our particular objective is to try to develop a line of continuous-feed machines in order to reduce manufacturing time and labor costs in the tannery.

This being the Fiftieth Anniversary of your Association, I was asked to speak on machinery developments during the past fifty years. It is an unfortunate fact that very few new machines have been developed during this period. Of course, there have been many improvements made in the old machines; but, very few entirely new machine concepts. The biggest period of tanning machinery development was in the last thirty years of the nineteenth century. Most of the tanning developments since that time have been in the field of chemistry, a field with which all of you are thoroughly familiar. I shall deliberately try to avoid any mention of matters chemical in this discussion, as I know nothing about this subject, even less than I know about tanning machinery.

Now, it is interesting to contemplate the reason for this dearth of new machine concepts during the past fifty years. By the very nature of their market, tanning machinery manufacturers are not among the industrial giants. The field for their products is too small. The manufacture of tanning machines does not lend itself to a production line basis. Also, these manufacturers have always to compete with an active second- and third-hand resale market of old tanning machines, which, in most respects, differ but little from the current new models. So, the total annual take of the tanning machinery manufacturers, as an industry, is small. Accordingly, the funds available to them for research and development are correspondingly small. Research and development cost money and the necessary funds just have not been available to the tanning machinery manufacturers. New machine concepts as a rule are the result of research and development. No research and development—no new machines. It's as simple as that.

*Presented at the Golden Jubilee Meeting of the A.L.C.A. in Cincinnati, Ohio, June 9, 1953.

The market for shoe machinery also is comparatively small, although there are two or three times as many shoe factories as there are tanneries. Still, in comparison to the market for automobiles, or electrical appliances the shoe machinery market is very small. However, the leading manufacturers of shoe machinery distribute the greater part of their production on a lease basis. This lease system, first used in the shoe industry to introduce the McKay Outsole Stitching Machine at the time of the Civil War, provides the steady income necessary for continuous research and development. In effect, the shoe manufacturers have been financing the research and development programs which the machinery manufacturers have been conducting for them. It is my belief that, had this been the custom in the tanning machinery business, there would be a great deal more really modern machinery in use today. The fact remains, however, that very few new machines have been developed in the past fifty years. There is a crying need for some modern high production machinery. To be of real value to the tanner, new machines must do either one of two things: Increase production appreciably, or improve the quality of the product.

Quite apart from machinery and chemical developments, there have been real improvements made in tanneries since the turn of the century. There have been improvements in working conditions, and in handling means. There is still room for improvement, particularly in regard to handling. The tannery of 1900 or 1903 was, I am told, a dirty, dark, damp, foul-smelling place. There were virtually no mechanical means of handling hides and skins, wet or dry, from one operation to the next. Wheelbarrows were the order of the day. Handling wet hides is a back-breaking job. The use of overhead cranes in the beamhouse and in the tan yards is a tremendous step forward. Electric tow trucks and fork lift trucks represent another important step in the right direction. In more and more tanneries, conveyors, of one sort or another, are commencing to put in an appearance—conveyors of the monorail type, and rubber belt conveyors. Of course, none of these—overhead cranes, electric trucks, conveyors—can literally be classified as tanning machinery, but they do represent mechanical progress, and progressive thinking. Another example is the modern method of forcing fleshings, and hair, through pipes, by means of powerful pumps and blowers, to the point in your tannery where you want them deposited. Progress has been made, of course, by means of industrial engineering, in plant layout. It is difficult in the case of the average tannery to draw up a really efficient flow chart because of the variety of the leathers being produced. If more than one type is being made, some backtracking is inevitable. Nevertheless, it is possible, through careful and systematic planning, to rearrange plant layout and machine locations in a manner that will cut down overhead expense to a tremendous extent. Good examples of this are certain comparatively new tanneries which are laid out in the shape of a U, and all on one floor. I've seen three of these. A certain midwestern tanner has completely renovated

and rearranged his tannery since World War II. The working space has been cut from five stories to two. New machinery and equipment have been substituted for old. As a result, with no loss of production, the pay roll has been cut from 835 persons to 375. And this in spite of the fact that there were no radically new machines, but only old machines redesigned to bring them up to date.

The two big developments in tanning machinery during the past fifty years have been, in my opinion, the introduction of individual electric motors in place of overhead shafting and belts; and, the use of hydraulics. But, here again, these developments do not really represent new tanning machines.

The transition to individual motor drives, though representing rather high initial costs, shows real advantages machine-wise, safety-wise, in a saving of space, and as a rule, in a saving of power consumption.

The use of hydraulics also contributes towards safety and towards economy in machine maintenance, as well as easier and more accurate control of pressures, and improved working conditions.

Other important developments are the trend towards enclosing all working parts, and better guards; and, centralized lubrication.

Let me point out again, however, that all of these developments are being applied to old machines, and that they do not represent new machine concepts.

Another important step is the introduction of humidity controls in the dry departments of the tannery. By the judicious use of humidifiers and air conditioning, the static electricity situation, which has always been a problem in tanneries, can be substantially improved, and excessive shrinkage of leather in dry periods prevented. Of course, the elimination of overhead shafting and belt drives in favor of individual motors also can be a tremendous help in licking the static electricity problem, especially when taken in conjunction with humidity control. But, humidifiers and air conditioning systems are in use in many industries, and can hardly be classified as tanning machinery.

Other real improvements have been made in the lime yards, by means of mechanical handling, agitation, heating, mixing, and conveying of the lime liquors. A great deal of hand labor has been eliminated by these means.

Broadly speaking, there are two basic types of machines in general use in tanneries. First, there is the full-width, helical-bladed, two-pass machine, which is used for fleshing, unhairing, scudding, setting or putting out, and wringing. The basic concept of this machine dates back to the last century.

The second type of machine can be referred to as the jack type. In this classification come such operations as staking, glazing, and rolling, where the machine works on only a narrow strip of the leather at a time.

In operating either of these types of machines, a certain amount of skill is necessary; and, also, hard work, in varying degrees. I am absolutely confident that, sooner or later, all of these operations will be performed on continuous-

feed machines—machines which will take the skill out of the operation, and some of the back-breaking labor. And, I can visualize conveyors taking your hides and skins, or leather, from one operation to the next.

Now let's go through the sequence of the more important tannery operations, and see just what changes have taken place in the past fifty years.

In many tanneries the first operation is to wash the hides and skins, and open them out, in wooden drums, or wheels, or mills. Other later operations often are performed in similar drums—stuffing, extracting, pickling, chrome tanning, fat-liquoring, coloring, etc. The drum might well be classified as a tanning machine. How has it changed in fifty years? Darned little, as far as labor or production are concerned. The driving methods have been improved, of course, and there have been improvements in the manufacture of the drum, and experiments with glass linings, and stainless steel. But the fact of the matter is that you're still using the drum; there has been no radical new invention to speed up the process. In many tanneries now drums are being loaded from overhead, sometimes from mechanically operated buckets. That's a real improvement in handling. But, virtually, you are using the same old drums. This goes, too, for vats and pits. Lime reels were being used as long ago as 1875. To be sure, they were cranked by hand, at first, but the method is the same today, only the drive is different. As far as I can determine, the use of overhead cranes is a development of the Twentieth Century, and, as I said earlier, this really represents progress, and accounts for a real saving in labor and accelerated production.

How about the rocker yards in the sole leather tannery? There's nothing new about rockers; they've been in use for eighty years.

I know of no startling new method of trimming or cropping in the sole leather tannery; nor, for that matter of trimming side leather, either in the hide cellar or upstairs. Nothing has taken the place of knives and shears operated by hand. The exception to this is the fact that there *are* good modern machines for trimming finished calfskins and kid- or goatskins. These come as floor models or table models. These are really modern machines. But we're getting ahead of ourselves.

We've already touched on the unhairing and fleshing operations. Here, the operator throws a wet side or skin into the machine, treadles the machine, pulls the side or skin out, turns it end for end, throws it back in the machine, treadles the machine again, pulls out the skin or side for the second time, and places it on a horse or pallet, or throws it in a pit or vat. The first fleshing machine of this cylinder type was invented by Mellen Bray of Maine in 1850. A fleshing machine invented by T. Roberts of Lynn, Massachusetts, in 1876, and improved by G. W. Baker of Wilmington, Delaware, had a lever motion, much like the present-day glazing jack. The present machines go back to the principle of the first machine but incorporate gripping rollers, and spiral blades in the cylinders. The present multiple-table unhairing machine was

patented in 1904 by William B. Turner and is a development of the machine invented in 1850 by Mellen Bray.

As far as I know, no continuous-feed machine for fleshing or unhairing has ever come on the market. We are working on such a machine at our research laboratories in Beverly, Massachusetts. The machine has undergone tannery trials, with not inconsiderable success, and may be close to the stage where it is ready for adoption for commerical use. If our plans meet with success, these machines will result in a really tremendous saving of labor in the beamhouse.

The first mechanical wringer was simply a brass roller weighted down and rolled over the hide or skin by hand. This was in general use until about 1875. The present type of wringer which is in general use utilizes felt covered rolls and is, like the fleshing and unhairing machines, a two-pass machine. Many sole leather tanners have converted these to continuous-feed machines, or built their own continuous-feed machines. Sole leather is heavy enough so the problem of wrinkles is not encountered to the same extent as with upper leather. There is a new continuous-feed wringer for upper leather on the market. At this time there are not many of them in use and I am not prepared to comment on the likelihood of this machine receiving general acceptance by the upper leather industry. If this new design is completely successful, it represents a very important step forward.

The first automatic setting machine was invented by F. A. Lockwood of Boston in 1876. This did not bear much resemblance to the setting or putting out machines in use today, which resemble the fleshing machine. They represent another example of the two-pass machine. Sooner or later this operation, too, is going to be performed in a continuous-feed machine. If our experimental continuous-feed unhairing and fleshing machines turn out to be the success we hope, we believe that it may be feasible to adapt this principle to setting and putting out.

The first splitting machine with a reciprocating knife was invented in 1809. The "Union Splitting Machine" was perfected in 1856. In this type the hide is drawn between a roller and a fixed knife. Some of these machines are still in use in calfskin tanneries for head-splitting or cheeking. A very good modern machine has been introduced into this country from Germany since World War II and is replacing the old Union splitters in calfskin tanneries. This machine utilizes a band-knife running in a vertical plane, and is a fine, high-production machine. A few years ago we put some effort into designing a high-speed machine for cheeking calfskins. We met with some success in our experiments, but also ran into several snags, and since the potential market in the calfskin industry would amount to about thirty machines, we decided to discontinue our efforts in this project in order to concentrate on developments of greater significance.

The horizontal band-knife splitter which is in general use in side upper leather tanneries was invented in 1883. It's a good machine, and it's a con-

tinuous-feed machine, which will lend itself to high production and maybe to conveyor systems some day.

Shaving follows the splitting operation in the typical side leather tannery. There have been many improvements made to the shaving machine since its introduction, such as increasing the width to the present popular 24" model, and substituting a power-driven bed roll for the loose roll. Shaving blades are still a problem. The operation still requires skill, and a full width, continuous-feed machine has not yet put in an appearance. I believe that, some day, there will be such a development. Incidentally, some years ago, we patented a method of shaving and splitting leather while frozen into a flat slab. Nothing ever came of this development.

In the book, "Modern American Tanning", published in 1902 by the Jacobsen Publishing Company, I find a picture of a shaving machine which bears a remarkable likeness to those in use today. This is in an advertisement of John Evans' Sons of Philadelphia, "Inventors, Patentees and Builders of Leather Working Machinery."

A few years ago we tried an interesting experiment in connection with this shaving operation. This consisted of an integrally cast cylinder and blades. These were made out of stellite. Our idea was that the longer life in this material, taken in connection with the time saved through not having to reblade cylinders constantly, would show the tanner a substantial saving. Actually, we found that under optimum conditions, these cast cylinders had a life about 25 times that of the average shaving blades; but it was practically impossible to find such optimum conditions. The operators in the tannery insisted on grinding the blades almost as often as they do the regular blades; and, the operators showed serious objection to the drag set up by the stellite. This is just another example of a research project which was not successful.

Now, when it comes to drying upper leather, real progress has been made in the past 50 years. Toggling is a development of a method first patented in 1904. Pasting has come into general use since then. The modern pasting system is truly an up-to-date tannery development, perhaps the most noteworthy development in the way of tannery machinery during this century. Attempts have been made, and experiments are still being carried on by my Company and others, aimed at faster drying times and elimination of the nuisance and expense of paste. Without any question, there will be improvements made in this direction. Many of you are familiar with our freeze-drying experiments. We stopped work on this project two years ago when we came to the conclusion that the idea was not economically practical. We did produce some beautiful leather by this method, leather which required no staking, and without the use of paste.

With regard to the drying of sole leather, there have been no new developments, although the tanners have added to their knowledge with regard to temperatures, and humidity control. Loft drying is still the order of the day.

One new, modern machine which deserves mention is the skin washer. This is a continuous-feed machine designed to remove the paste from sides or skins, and also serves to wet them back for subsequent operations.

Staking. The old belly-stakers and clamp-stakers are still in general use. This primitive machine which works on only a small strip of the leather at a time is slow, expensive to operate because of the skill involved, and dangerous to the operator. Many attempts have been made to introduce a full-width machine. While some of these are in use, none of them have proved successful to the point where they can be used on a great variety of leathers. To be successful, such a machine must have a selective action in that it will operate more vigorously on the hard parts of the leather than the soft. We have been working on such a development for several years, and our experimental full-width, continuous-feed machine has undergone tannery trials with some success; but, I can't truthfully say that, to date, we can satisfactorily stake all types of leather.

Now, the conventional pendulum-type sole leather rolling machine is about as antiquated a contraption as can be imagined. It has all the disadvantages which I have just mentioned as applying to the old staking machines. Various attempts have been made to design a full-width, continuous-feed machine. We have such a machine, still in the experimental stage, which has been undergoing tannery trials for over 2 years. This machine will wet roll or dry roll crops or bends, with a 3-to-1 production advantage over the old pendulums; but, due to the very small market for such a machine, which means a high manufacturing cost, and the fact that the development cost has necessarily been large, this is an expensive machine.

Most upper leather now is buffed on one side or the other, or both. For this operation there is a modern machine in the full-width machine made by at least two manufacturers. In addition to being full-width, this machine also is, in effect, continuous-feed, although it is usually necessary to insert a small part of the side or skin into the machine, and then pull it out and turn it end-for-end before allowing it to pass all the way through. As a rule, the machine is set up to return the completely buffed leather to the operator. These machines are now in widespread use, but have not completely replaced the older narrow type machines, many of which are used for spot buffing. I understand that the modern full-width machine is so accurate that it is possible to buff the printing off a piece of newspaper without destroying the paper. That's a good machine.

After buffing or snuffing, it is necessary to remove the dust from the leather. Progress has been made in this operation. Not too long ago almost all upper leather was dusted in the old box brusher, a slow and laborious operation. then, along came continuous-feed, high-speed brushing machines, and the latest development is our USMC Air Blast Dust Removal Machine—Model A. This is a continuous-feed machine which utilizes compressed air rather than brushes to clean the leather. With a feed speed of 200 feet a minute, I

believe that this is the highest production machine in general use in tanneries today. Practically all the side upper leather produced in this country today is cleaned in this machine. There are about 90 of them in use, which gives a good indication of just how hungry the tanners are for really new machines, considering that we adopted this machine only 4 years ago.

We have in the development stage a stacking machine which will take the leather from this dusting machine and stack it on horses. This stacker can be used also behind a skin washer, or a spray seasoning conveyor, or any other continuous-feed machine, as more come into use.

Seasoning machines are a development of the Twentieth Century, and represent real progress towards conveyorization. Spray seasoning with conveyors running through drying tunnels represents a further step in the right direction.

The boarding machine, while fairly modern in its concept, was originally patented in 1873, and improved in 1885. This is a full width machine, and continuous in its feed, bringing the work back to the operator, which is necessary for a 2-way or 3-way boarding. It is a good machine production-wise, but hardly new.

The glazing jack which we mentioned briefly earlier is certainly a primitive machine, but the effect which it produces on the leather apparently can be obtained by no other means. Here again, I believe that a way will be found some day to get the desired effect by means of a full-width, continuous-feed machine.

The heavy presses in use for plating and embossing may represent good machine design, but it is certainly a slow operation. I can't help but feel that a way will be found to speed it up, probably by means of continuous-feed machines.

The measuring machine in general use in this country is essentially the same as one patented in 1883 by W. A. Sawyer of Danversport, Massachusetts. It is a modern machine in that it embodies the continuous-feed principle. However, it is not as accurate, nor as fast, as the pinwheel machine which is in general use in Europe, and it requires more maintenance than the pinwheel. Our American machine has a tendency to over-measure. This fact is well known, but, if any one tanner changed to the pinwheel machine, he would have to up the selling price of his leather by perhaps 2%. So, they all stick to the same old machine. There have been various attempts to market measuring machines embodying an electric-eye principle. It is possible, under ideal conditions, to measure accurately by this method, just as accurately as with the European pinwheel machine; but, from what I've seen, there would be no advantage production-wise. Either the so-called "Korrect" machine, or the pinwheel machine, could be adapted to a conveyor system. We have done some work on a device to stamp the measurement on the leather automatically after it has passed through the machine. This might be used

in conjunction with our stacker to dispense with the need for the man taking the leather away from the measuring machine.

We come now to the shipping room, and I hope that, one day, we shall see bundling machines in operation here, to do away with the hand labor involved in wrapping the leather with paper and string.

I have not, of course, covered every single tannery operation, but I have tried to demonstrate what I said at the start; that the biggest period of tanning machinery development came in the 30 years just prior to 1900, and that the number of real innovations since that time has been disappointing. To enumerate the really new machine concepts, they are: Trimming Machines for kid and calfskins; a band-knife cheeking machine; the Pasting system of drying, and the skin-washer which goes hand-in hand with it; the full-width buffing machine; dust removal by air blast, and seasoning machines with conveyors.

I think that you chemists are way ahead of the machinery manufacturers, and I hope that the next 50 years will see us starting to catch up. Unless, that is, during that time you make discoveries which will make all tanning machinery unnecessary.

Received July 6, 1953.

Leather Oils During the Past Fifty Years*

T. A. Faust

When this Association met in Cincinnati in 1927 I read a paper entitled 'Fifty Years Ago'. This paper was an abstract of the book which Jackson Schultz wrote in 1876 namely 'Leather Manufacturing' and I expressed surprise that Schultz wrote about so many things that we thought were comparatively new.

Now on the 50th anniversary of this Association I have been asked to prepare a paper on developments in leather oils during the past half century. Perhaps this entitles me to be called 'The A.L.C.A. Fifty Year Man'. In order to get a picture of leather oils in 1903 I wrote to my old friends who were active then and I secured great help from them, including some facts about what was used prior to that time, some of which may interest you.

In the book written by Schultz in 1876 he mentions sod oil or degras, also tallow and oil probably cod or perhaps whale oil; these were the generally used materials, but I was surprised that sod oil or degras was used so long ago. Incidentally I have spent a lifetime trying to find out what a tanner means when he says degras; does he mean wool grease, or moellon, or sod oil which is a type of moellon? However, I believe Schultz refers to sod oil. Mr. Trask wrote me that prior to 1901 sod oil and also wool grease was imported from England.

* Presented at the Golden Jubilee Meeting of the A. L. C. A. in Cincinnati, Ohio, June 9, 1953.

I am indebted to Caspar Drueding for information on the early production of moellon in the United States. His company started tanning chamois and making moellon in 1882; only one grade was made, pure chamois moellon containing 25% moisture.

The oil was kicked or fulled into the skins, and after tanning the skins were soaked in warm water and pressed. This first pressing was moellon. The skins were then drummed in a sodium carbonate solution to emulsify the oil and were pressed a second time. This oil was acidified with sulphuric acid, skimmed off and boiled down to about 5% moisture and was sold as sod oil, often miscalled degras. Incidentally the same procedure was then used in England, France and Belgium.

Moellon is made a little differently today and I will described this later.

Mr. Charles Morrison Sr., has given me information on oils used prior to 1900. Tanners were using mostly cod oil and some fish oil on their sole leather and belting butts. However, he started to use mineral oil about that time and he also mentioned that even before this, one company was selling hard grease for harness leather and that this developed very fast. However for heavy leather, sod oil, fish oil, and tallow were the principal materials used.

Apparently sod oil was used on lighter leathers before 1900 but for calf and kid, fat liquors made with fig soap and either cod or neatsfoot oils were largely used. Lamb in his book published in 1907 wrote that sod oil was then used for light leather in England. Egg yolk was used on kid leather together with flour, neatfoot oil and some soap.

Moellon emulsions were used both on skiver's and hatter's leather but around 1912 when the demand came for dryer leather, sulphonated oils were added to the mixture; in fact at that time three different types were used, moellon emulsion, sulphonated cod oil, and mixtures of moellon and sulphonated cod; these were used both on vegetable and chrome re-tanned skivers and sheep. My information on side leather procedure is not too accurate but apparently most of it was hand stuffed in those days rather than fat-liquored as it is today. Harness leather was wheel stuffed but shortly afterwards dry dipping became popular. Mr. Yocum used the dry dip method on belting butts about 1909 and eventually persuaded my people to adopt this method for harness leather, and shortly many harness leather tanners and nearly all the belting butt tanners used the dry dip method. In this method, the leather is dried to almost absolute dryness in a hot room, dipped into very hot grease, then into warm water and dried in this hot room, the temperature of the room being dropped slowly over a period of say twelve hours.

There were some seven or eight developments during the past half century but the most important was that of sulphonated or more properly sulphated oils.

The first production of sulphated oils goes back more than a century. The colour turkey red originated probably in Turkey several hundred years ago

and the process was brought to Scotland before 1800. The process was applied to cotton, it was based on oiling, mordanting with alum, and dyeing with madder, the dyeing principle of which is alizarin. The oiling referred to is rather vague but I understand emulsified olive oil was used.

Burton & Robertshaw in their book 'Sulphated Oils and Allied Products' write as follows:

> "Sulphation started in 1831, when Fremy studied the effect of concentrated sulphuric acid on olive oil, oleic acid and almond oil, but A. Bunge was the first to prepare a sulphated olive oil in 1834. He used this as a mordant in dyeing calico red and thus it was the forerunner of the modern turkey red oil."

Incidentally the authors of this book state that sulphated oils are remarkable not only for variety of materials which can be sulphated but also for the variety of products which can be obtained from one oil; also that a sulphated castor oil containing a high percentage of Combined SO_3 was made as far back as 1911 by sulphonating with chlorosulphonic acid in the presence of a solvent, and that marine animal oils were sulphated since 1910. They also mention sulphated neatsfoot oil. However, it was about 1875 that castor oil was first sulphated both in Germany and England; this is the turkey red oil as we know it today.

Turkey red is a very vivid red of great fastness and was used particularly for red tablecloths and bandana handkerchiefs. I was anxious to get a cloth of genuine turkey red and wrote Mr. Horner of the CIBA Company who replied in an amusing letter saying 'Where on earth could I find a cloth dyed the old turkey red? and then answered his question by saying—'Why in England'. So he wrote to the English office and later received from them a cloth dyed in 1898 and I have here this cloth which is still after fifty-five years a vivid bright red. I also have a second pattern illustrating turkey red in a print-on style and I thought you would like to see these cloths. The emulsified oils used originally in dyeing this shade were superceded about 1850 with the sulphated castor oils, i. e. turkey red oils and these oils are the basis of the sulphated oil industry.

It was thought that Proctor first used turkey red oil for fat-liquoring leather in 1890 but according to Lamb, turkey red oil was used in those days as a dyeing assistant, not as a fat-liqour. However a little sulphated castor oil was used in leather around 1900.

Mr. Charles Gulick gave me considerable information about the start of sulphonation of oils for leather. He made sulphonated castor oil for the textile trade beginning about 1905. A few years later he and his associates decided to work on the possibility of making sulphonated oils for leather, using cod oil; they did this since cod oil was largely used and thought that if it was solubilized, it would be more efficient. They treated cod oil somewhat

similarly to the way that they had been sulphonating castor oil, had some tests made in the Newark tanneries and obtained results so encouraging that they concentrated on producing sulphonated cod oil for leather. I think we can assume that this is the beginning of sulphated oils in the United States. You will note I am using the terms sulphated and sulphonated interchangeably; the correct nomenclature is sulphated but commercially they are called sulphonated and it was many years before chemists realized these products were sulphates and not sulphonates.

However very shortly others started working on the same idea; John Yocum about 1912, F. S. Walton the same year, Mr. James S. Bent about 1911 and Mr. Mark Smith about 1913. The first sulphonated oil in Canada was made by T. A. Faust in 1915.

In 1912 our Journal printed an abstract of an article that appeared in 'Gerber' written by Eitner. He spoke of tanning oils which possess emulsifying properties, used to assist in the actual tanning. They were apparently elaine or red oil soaps mixed with mineral oil but added that for chrome tannage these soap mixtures are decomposed in the liquors and sulphonated oil should be used. He spoke of alkaline oils for quick tanning processes to help dissolve Argentine extract but they must only be used in moving liquors in the rockers, not in layers, and mentioned a bleaching oil which was 30 per cent sulphonated fish and 70 per cent mineral oil. He wrote that the sulphuric acid is in an organic combination and need not be feared.

Now this article was written in Germany in 1912 and there are some tanners who use sulphonated oil in their extract drums today although not as a tanning oil which was really Eitner's idea. There are on the market today some bleaching oils. Apparently Eitner had been working for years on sulphonating fish oil using the information obtained in the sulphonation of castor oil as the basis of his experiments.

The sulphonation of oils for leather developed very rapidly in the U. S. from 1912 to 1920. Originally sulphonation was done in crocks but later it was conducted in large leadlined sulphonating kettles and still later in either monel metal or stainless steel kettles all equipped with the necessary cooling apparatus. There are some eight or nine large manufacturers of these oils in the States and five in Canada.

In addition to sulphonated oils there have been other developments in emulsifying oils. First; sulphonates of the naphthenic type mineral oils formerly known as mahogany or mineral oil sulphonates were developed some thirty years ago. They are made from the petroleum sulphonic acids produced in the acid refining of petroleum when making mineral oils. These products are the true sulphonates and not the sulphates as the so-called sulphonated oils actually are. One company produced these oils around 1914 and during the last twenty years they have been used for emulsifying mineral oils and sometimes fatty oils, and many sulphonators use some of these sulphonates to produce special types of fat liquors.

Another recent development is the production of non-ionics; they were originally produced in Germany by I. G. The earlier work in the U. S. was done about seven years ago and today several manufacturers are producing them. For some work they are superior to sulphated oils and often used with sulphated oils to get unusual results on such leathers as calf and light sides.

Cationic oils might be mentioned; they are not miscible with sulphated oils and are not largely used on leather. An additional product used to a small extent, anionic in character is sulphated alcohol.

An important development is the improvement in mineral oils and waxes for leather. I mentioned that Mr. Morrison Sr. used mineral oil back in 1898, but Dr. Koppenhoefer gave me some interesting information namely that in 1887, the Vacuum Oil Company, then located in Rochester had pushed the use of mineral oils to curriers in that city who wrote a testimonial as to their value. This was only eight years after petroleum was discovered in Pennsylvania.

While the earlier mineral oils were not too successful, about 1900 several manufacturers improved these oils so that they made an excellent leather oil and replaced to some extent cod, fish, and neatsfoot oils. About 1912 many sole leather tanners adopted mineral oils although certain tanners never did use mineral oils until the second war when fatty oils became prohibitive in price.

As late as 1910 an article was published in our Journal expressing doubt as to the suitability of mineral oil for leather, saying they were used principally as an adulterant of cod oil and were not suitable for leather: the reason is amusing; since chamois couldn't be made with mineral oil he figured that cod oil is digestible whereas mineral oil is not, therefore the latter is not suitable; also that mineral oil is not emulsifiable and could not be incorporated into wet leather. Later he wrote that he was working on emulsifying mineral oil which would make the oil better for leather, saying that since cod oil and other fatty oils were required for soap, mineral oils should be used in leather. Nevertheless several producers were at that time, that is 1910 selling satisfactory products to tanners.

Also in 1910 another article was published in our Journal on the use and suitability of mineral oils for leather; emphasizing that they had to be selected carefully, properly decolorized and the gummy portions removed. No single distillate was considered satisfactory, it was necessary to blend selected stocks to get a satisfactory oil and that the performance of oil in leather was to give pliability, tensile strength and water-resisting properties; also that mineral oil eliminated or at least reduced spew.

There are three different types of mineral oils, the paraffinic, asphaltic, and naphthenic but it was soon found that the asphaltic type was not suitable for leather and that the paraffinic type was much better. Recently several tanners oil manufacturers found that the naphthenic type has certain advantages.

Another development was the use of mineral waxes in admixture with tallow, stearine, wool grease, etc. to make leather stuffing compounds. This was pioneered in the early 1890's. Proctor mentions the use of paraffin wax for stuffing leather in his book published in 1903, emphasizing its oiliness, stability and particularly its non-spewing characteristics.

An interesting economic fact is that whereas years ago mineral oils were one-fourth to one-sixth the price of cod oil, today suitable mineral oils are nearly one-half the cost of cod oil. This is partly due to the recent low market on cod oils; the same applies to stuffing greases; paraffin wax, the basis of mineral stuffing greases was worth about one-third to one-fourth the cost of tallow, but today is selling for more than tallow.

There are several other products of lesser importance that might be mentioned, for instance about 1915 Carleton Ellis developed hydrogenated oils and at one time hydrogenated fish oil was used in stuffing greases, in fact it was a component of some of the mineral stuffing greases mentioned above; the hydrogenated fish oil could be brought up to a much higher melting point than tallow and therefore a higher melting point stuffing compound could be produced. However in view of the low price of tallow and oleostearine today no large tonnage of hydrogenated oil is now used.

As mentioned earlier, moellon or sod oil was used before the beginning of the century, and is used largely today particularly on heavy oiled side upper leather, but it is made differently now. Mr. Drueding advises that the present procedure is to add soda in the oil drum before oxidation takes place and after oxidation is complete to add more soda solution. The skins are then pressed or run through a wringer. The emulsified oil is then cut with acid, skimmed off, and boiled down to about 3 per cent moisture. This is called moellon and if 25 per cent moisture moellon is wanted, the moisture is put back in an emulsifier drum.

About 1910 it was found that by blowing cod oil with hot air, oxidation took place and a product similar to the natural moellon was produced. At first the tanner viewed with skepticism this artificial moellon and I remember about 1912 analyzing samples and the tanner's desire was to find if it was natural or artificial. We discovered that in the natural there were tiny hide fragments but it didn't take the manufacturer of artificial moellon very long to incorporate a little ground hide in his product.

There is a fair amount of artificial moellon produced in the United States and Canada although I believe the consumption of natural is greater than the artificial. At present the natural sells for less than the artificial.

Wool grease should be mentioned; it is used particularly on side leathers intended for heavy army boots where a fairly heavy stuffed leather is required. Wool grease has been used a long time but since the first world war it was discovered that certain of its characteristics are of extreme value in leather, namely that when incorporated in a stuffing mixture, it preserves the unctiousness of the mixture; that is it enables the leather to remain soft

and pliable in Arctic temperatures and yet in the Tropics, the leather remains pliable not oily; the wool grease seems to enable the stuffing compound to stay put in the leather. During the last war, strong efforts were made to work out a substitute for wool grease but I doubt if anyone entirely succeeded.

Wool grease is recovered from the wool of the sheep and is entirely different from sheep grease which is recovered from the skin of the sheep. Sheep grease is used also for leather but is regarded as a poor alternate for neatsfoot oil; it is usually pressed but it does not sulphonate too well and is not very highly regarded by most tanners; it frequently contains some solvent left over from the degreasing process.

Brief mention might be made of certain other oils; mustardseed oil was used as an alternate for neatsfoot oil when the latter became prohibitive in price, and other seed oils were also used. These oils have a low pour point and so depress the pour point of neatsfoot when added in small percentages. Cocoanut oil is used as an ingredient of fat liquors for white leathers; this is a development within the last twenty years. While cod oil is the standard marine animal oil, other oils have been used; for instance manhaden oil, and Yocum used this oil at the beginning of his work on sulphated oils.

Egg yolk was apparently used before the beginning of the century and is is still used to a considerable extent in fat-liquoring kid leathers and I believe even on sheep. Most of the egg yolk is made from hatchery infertiles; today however, egg yolk substitutes are made, usually with lecithin as the base, derived from soya bean oil refining.

Finally one group of products must be mentioned. Doctor Kennedy of the Quarter Master General's Office in Washington told us that it was necessary that leather chemists develop synthetic oils and fats similar to the ones that were devised in Germany during the last war. Research work was done at Ohio State University by Dr. Brown and others on synthetic fat replacement materials for leather; some of the work in conjunction with the Tanner's Research Laboratory.

I believe Mr. Meo is going to discuss the chemical nature of the synthetic fats and I will confine my remarks to the economics. As you know, most oils and fats are extremely low in price; in fact they were about the first commodity to take the grand nose dive from the inflated valued of say five years ago. This rapid deflation was caused by two things:—First—the excessive prices of most oils and fats; neatsfoot oil at 40 cents and tallows around 17 cents a pound— values which were out of proportion to some other commodities.

Second—the decline of oils and fats was greatly accelerated by the tremendous increase in the manufacture of synthetic detergents most of which use petroleum as the base. Today soap manufacturers are selling much less soap and much more syndets as they are called. This has depressed the price of grease and tallows; the differentiation being that tallow is beef or sheep

fat and grease mostly hog fat. Tallow and grease producers are worried about this, and are contemplating research to develop other outlets, in fact the Eastern Regional Laboratories has already published a pamphlet on the problem. Latest figures indicate sale of syndets were about three-fourths that of soaps last year.

The low price of tallow depressed other oils and fats with a few exceptions such as wool grease which sold at extremely high prices last year due to reduced scouring of raw wool, but in the past few months has dropped considerably.

Most vegetable oils however came down to quite low levels but during the past few months some have been advancing. Incidentally I am confronted with such changing markets that some of my statements will not be correct for any length of time.

However we must face the economics of the synthetic fat suggestion by Dr. Kennedy. Under present conditions synthetic fats in my opinion are out of the picture but one never knows what may happen, and it is possible that in the future these synthetic fats might again be of interest. At this moment there is not much indication that such will be the case in view of the high production of oils and fats not only in North America but through the entire world, and the lowered consumption by the soap industry. Of course depressed values have a habit of reducing production and again increasing prices.

In conclusion, I believe the American tanners are well satisfied with the supplies, quality and working characteristics of the various types of manufactured oils and fats that are being offered to them; of course further improvements will be made in these products and we can look for possibly new developments in the next few years.

As I mentioned earlier, I received great help from my friends and I wish therefore to thank the following who have assisted me: Mr. Chas. Gulick, Mr. Arthur Trask, Mr. Charlie Morrison Sr., Mr. Caspar Dreuding, Mr. Morrison Jr. and also Mr. Meo who secured certain information for me from Mr. Mark Smith. I also thank Dr. Koppenhoefer for information in mineral oils and Mr. Horner of the Ciba Company for his help.

ABSTRACTS

The Significance of Mucoids of Hides and Skins in the Limeyard. R. Reed. *J. Soc. Leather Trades' Chemists*, **37**, 75 (1953). It has long been known that mucoids are present in animal skin. From mucoids isolated from skin, as a clear viscous fluid, two compounds have been identified. One is hyaluronic acid, thought to be a polymer produced from a disaccharide unit containing acetyl-glucosamine and glucuronic acid. The other is chrondroitin sulfate, a polymer formed from a disaccharaide unit containing glucuronic acid and acetyl-galactosamine. It has been found that mucoid is distributed generally throughout the skin, the regions shown to be particularly rich in mucoid being the papillary (upper)

layer of the corium especially around the hair follicles, the junction of this layer with the epidermis and the vascular process in the corium. In fact all those sites in the skin rich in elastic tissue are also rich in mucoid. The function of the mucoids is not definitely known. There is evidence that it is both physical and chemical. Much of the evidence indicates that the chrondroitin component is an essential and integral part of the fibrous structures and that this material (and possibly also hyaluronic acid) is concerned in the cohesion and general chemical stability of the collagen fibrils. There seems to be little doubt that the mucoids absorb enzymes and bacteria thus preventing their disintegrating action on the collagen. It is suggested that, in any discussion of the mechanism of liming, removal of the mucoids with a consequent change in the cohesion of the remaining structures should be borne in mind. It should be possible to produce pelt satisfactory for tanning by removing the mucoids with mucolytic enzymes. It is also suggested that a suitable method of unhairing would be the complete removal of the epidermis after destroying the mucoids at the epidermal-corium junction with mucolytic enzymes. It is thought that possibly the difficulties encountered in the soaking back of dried hides arise from the inter-fiber fluids (mucoids) forming hard cementing substances encasing the fibers. This hard substance could be removed by the use of mucolytic enzymes. Much remains to be done before it can be definitely established that pelt of high quality can result from the mere removal of mucoid materials alone. Preliminary results are encouraging. These are discussed in the following paper. R.H.T.

The Unhairing of Hides and Skins without Lime and Sulfide. The Use of Mucolytic Enzymes. D. Burton, R. Reed and F. O. Flint. *J. Soc. Leather Trades' Chemists*, **37**, 82 (1953). One of the major problems of the leather trade is the question of the effluent of the lime yard. Disposal is a costly problem, yet the authorities are getting more and more critical of stream pollution. In the past liming was very slow, usually taking several months and having been known to extend to 18 months. The practice of sharpening rose from the desire to quicken the loosening of the hair. It is certain that in these long periods bacteria played a part although the present liquor containing sulfide, even though old by current standards, contains few if any bacteria. In the past fermented sour liquors, fermented barley flour, meal, egg yolk, animal brains and sour milk were used. While not as easily controlled as liming they were widely used and are still used in central Asia. The remarkable thing is that both acid and alkaline liquors were used for unhairing. If we can find what is common to both, the mechanism should be better understood. As has been brought out in other papers, all fibrous protein structures are in close association with mucoids. In skin it is particularly rich at the epidermal-corium juncture and around the hair follicles. It has been suggested that soaking back, unhairing and bating may in essence be the removal of the mucoid material. Research has shown that it may be removed by treatment with alkalis and by the action of mucolytic enzymes. This, seemingly, is the common property of the alkaline and acid liquors used for unhairing. With regard to the first, lime has been used for hundreds of years. It may also be significant that in nearly all methods depending upon proteolytic enzymes, pretreatment with alkali is essential. Preliminary experiments show that mucolytic enzymes will bring about unhairing. So far pectinase at pH 4.0, diastase at pH 6.5, and pancreatic elastase at pH 8.9 have been particularly effective. At room temperature fresh calf skins require 3 days. The epidermis is detached in a single sheet bringing all the hair with it. The hair appears to be undamaged. The grain surface exposed is of high quality. Distortion of the grain due to rapid swelling or plumping at high pH can be avoided. Since mucoids are distributed throughout the fibrous structure, corium mucoids should be removed by the mucolytic enzymes resulting in a bating action. This was found to be so. It was, however, found

that much material could be extracted from the unhaired pelts by soaking in 10 per cent NaCl at room temperatures for a few hours. It seemed that the corium-flesh layer was also attacked because the soft fleshy layer removed easily. After soaking in common salt the hide was pickled over night and chrome tanned as usual. The grain was smooth, did not crack on flexing and had a pale even color which may be an advantage in making light colored leather. The pelt could be fatliquored and dyed in the usual manner. In view of the results it is apparent that unhairing is done by both alkaline and acid liquors, when the acidity is attained by fermentation, because both remove the mucoid material which plays an important part in the cohesion of fibrous protein structures. All the fermented sour liquors naturally contained such enzymes as diastase and other amylases. Yeast would contain zymase and the fruit juices would contain enzymes similar to pectinase. While the research is still in its early stages, it seems probable that the process can be well used for light leathers. For sole leathers other factors, such as plumping, will have to be considered. R.H.T.

The Mechanics of the Process of Drumming. By R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **37**, 87 (1953). This is a theoretical consideration, mathematically developed, of what happens to a single hide and a pack of hides during drumming. The theory is discussed at some length. In the discussion it is emphasized that theoretical considerations can do no more than serve as a rough guide for drumming procedure. The investigations led to the following conclusions. A skin that is placed in a rotating drum without shelves or pegs cannot, if placed near the bottom of the drum, be carried sufficiently far up the ascending wall to cause it to fall. Instead it will merely slide backwards and forwards in the lower half of the drum. If the drum is rotating at a speed much higher than any used in practice, the skin, if it starts high on the descending wall, may be carried high enough to fall, but only if its passage through the lower part of the drum is unhindered by liquor or by collisions with other skins. At drum speeds similar to those used in practice it is highly unlikely that a skin can be carried high enough to fall, even if its passage through the lower parts of the drum is unhindered by anything more than the friction between the wall of the drum and the skin itself. The shelves or pegs of a drum are not merely auxiliary apparatus, whose function is to enable larger quantities of skins to be carried upwards and dropped at each revolution of the drum. They form an essential part of the drum without which no real drumming action can occur. R.H.T.

Some Factors Influencing the Tear and Tensile Strength of Split Hide Leather. G. E. Benskin. *J. Soc. Leather Trades' Chemists*, **37**, 126 (1953). The author presents experimental data to support the following conclusions. The tensile strength of the collagen in split hide leather is about 18 per cent greater than that of the pelt from which it was made. The tear strength is about 16 per cent less. The grain split should be at least 2.3 mm. in thickness. With leather of greater thickness than that minimum, the increase in tear strength is almost proportional to the increase in thickness. The variation of pH and salts in the range normally found in split hide tannage does not materially affect the strength. The two bath chrome process behaves in a manner similar to vegetable tannins in imparting an increase of about 18 per cent to the tensile strength of the pelt from which it was made, whereas the single bath process imparts no increase in strength. The tensile strength is not greatly affected by the extent of the grease concentration. The tear strength is governed to a large extent, more by the quantity of the grease present, than by the quality. Sulfated oils do not contribute to the tear strength quite to the same extent as straight or non-sulfated oils. The even distribution of the grease throughout the leather is of great importance in obtaining maximum tear strength. R. H. T.

Wear Tests on Leather Soles. C. D. Pomery. *J. Soc. Leather Trades' Chemists*, **37**, 145 (1953). Recent work in the U.S.A. has shown so little relation between abrasion tests and wearing properties that the Physical Testing Committee of the A.L.C.A. has recommended that these tests be discontinued. However these conclusions are unreliable because based on the false assumption that the rate of wear is a linear function of time. Thus the abrasion tests and measurement of wear have not been compared satisfactorily. The present experiment was designed to investigate the correlation between wear and laboratory abrasion tests for typical English sole leather. The comparison was made on 10 sole leathers from different tanneries using laboratory abrasion tests and by actual walking wear on a concrete surface. A full description of the methods of sampling and means of compensating for irregularities in walking habits is given. Laboratory abrasion was measured on four samples cut from around every sole using a rotary abrasion machine with two different loads. From all soles before tests 1½-2 mm. grain split was removed in hopes that this would cause the most uniform portion of the sole to be used during tests. The walking track was of clean, dry concrete, oval in shape and 35 yards in circumference. The results of the experiment do not condemn abrasion tests but give some evidence that the rate of wear and abrasion are related. The difference between the leathers tested was too small for accurate comparisons to be made, and the measurements of abrasion gave a much larger variation, with change in sampling position, than was expected. The abrasion tests, as carried out at present, appear to distinguish between very good and very bad leathers, but not between leathers closely similar in quality. The tests become unreliable because of sampling fluctuations which are serious causes of error when small samples are used. In this experiment, however, the abrasion tests did distinguish between the leathers as well as did the wear tests. For further study the measurement of both rate of abrasion and rate of wear must be refined so that a more accurate comparison can be made between the leathers in spite of sampling variations.
R. H. T.

Opacities of Water Pigment Finishes. H. F. Vollmann and J. Kendall. *J. Soc. Leather Trades' Chemists*, **37**, 154 (1953). This is a critical comment on the electrometric method for measuring opacities of water pigment finishes by Mudd and Downs [*J.S.L.T.C.* **37**, 67 (1953), *This Journal*, **48**, 519 (1953)] A comment on the criticism by Mudd and Downs is printed as a footnote. R. H. T.

Improved Method for Determining "Free" Sulfate in Chrome Liquors. S. G. Shuttleworth. *J. Soc. Leather Trades' Chemists*, **37**, 156 (1953). The standard method for determination of non-complex bound or "free" sulfate in chrome liquors is by precipitation with benzidine hydrochloride followed by filtration and titration with standard alkali. Due mostly to filtration troubles this method is both tedious and inaccurate. The following method using a centrifuge is recommended. Use 12 to 15 ml. centrifuge tubes of elongated shape (6 x ½"). Add 5 ml. of chrome solution of about 0.1 per cent Cr. strength. Add approximately 5 ml. of benzidine hydrochloride solution (15 g. benzidine and 50 ml. conc. HCl boiled and diluted to 1 liter). Shake or stir. After 1 min. centrifuge at 6,000 r.p.m. for 3 min. Pour off liquor. Add 10 ml. of water and stir. Centrifuge at 6,000 r.p.m. for 5 min. Repeat this washing once more. Transfer the precipitate to a 100 ml. evaporating dish and titrate with 0.02 *N* NaOH using phenolphthalein as indicator, completing the titration at boil. The usual table model centrifuge will handle two pairs of duplicate determinations and the total time averages 15 min. per determination. R. H. T.

The Durability of Upholstery Leather from Split Hides. R. F. Innes, M. H. Mayer and R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **37**, 158 (1953). Earlier work on the

deterioration of leather in polluted atmospheres is reviewed. In the past interest has largely centered on deterioration of book bindings. Up to 1945 experimentation seemed to justify the following statements. (1) Absence of salts tends to increase rate of decay. (2) The presence of salts exerts a protective action. (3) Absence of grease does not favor decay. (4) Catechol tanned leathers free from salts are more liable to decay than pyrogallol tanned leathers free from salts. The experiments described were designed to test three previously developed methods for measuring deterioration. They are the gas chamber method, high pressure bomb method and application of acid and hydrogen peroxide method. Some modifications of the first are described. The experiments were performed on leather from a commercial catechol tanned split hide. Prior to tests, salts were added to the leathers as follows: none, aluminum sulfate, aluminum sulfate on degreased leather, sodium chloride, potassium lactate, calcium oxalate, ferric sulfate followed by calcium oxalate and, lastly, calcium oxalate followed by ferric sulfate. The results show that the bomb and peroxide tests arrange treated leathers in the same order of merit, but the gas chamber gives a different order. It is concluded that there is a satisfactory agreement between the physical and chemical tests of deterioration except in a few instances. Measurements of soluble nitrogen and tensile strength lead to similar conclusions. Present indications are that the gas chamber tests simulates natural conditions most closely. If the gas chamber test is so accepted and losses of tensile strength are accepted as the measure of deterioration, protective treatment falls into the following order of merit: alum (best), lactate, oxalate, chloride, none (worst). As judged by the chemical results the order of merit is: alum (best) chloride, lactate, oxalate, none. R. H. T.

Nomograph for Use in Determining the Acid Figure of Leather. G. A. R. Taylor. *J. Soc. Leather Trades' Chemists*, **37**, 180 (1953). A nomograph is presented to avoid the tedious arithmetic work entailed in calculating the logarithm of dilution when determining the acidity of leather by the method of Atkin and Thompson. R. H. T.

The Mechanism of Vegetable Tannage. R. O. Page. *J. Soc. Leather Trades' Chemists*, **37**, 183 (1953). In view of the practical and theoretical importance of hot-pitting, an examination was undertaken of the retanning of vegetable tanned leathers at temperatures in the neighborhood of the shrinkage temperature of collagen. Delimed cowhide butts were tanned for six weeks in gradually increasing concentrations of wattle until a final value of 12 per cent concentration of total solids was reached. At this stage the tanned pieces had, on a collagen basis, 32 per cent fixed tannins, 38 per cent combined water solubles and a shrinkage temperature of 82 degrees C. They were retanned for 48 hours in wattle extract containing 12 per cent soluble material at temperatures from 50 to 70 degrees C. In one series, at pH 4, the hot tanning was continued for nine days as well as for two days. A series at pH 3 was tanned only for the two day period. Tables I and II show the results obtained.

TABLE I.

RETANNAGE AT pH4.

Time of Tannage	Temp. °C	% Wt. Change	% Area Change	Shrink Temp. °C	Fixed Tannin	Combined Water-Sols.	Total Tannin	Acid Combined	Moist. Content
2 days	60	+ 1·3	0	81	56	39	95	8.7	33
—	65	−11·6	−10·5	—	72	25	97	8·4	34
—	70	−27·9	−38·0	—	87	10	97	8·6	32
9 days	60	−14·0	−10·0	—	103	13	116	8·5	36

TABLE II.

RETANNAGE AT pH3.

Time of Tannage	Temp. °C	% Wt. Change	% Area Change	Shrink Temp. °C	Fixed Tannin	Combined Water-Sols.	Total Tannin	Acid Combined	Moist. Content
2 days	50	+ 1·0	0	72	69	38	107	8.6	36
—	55	−18·0	−11·0	—	68	32	100	8·6	31

At pH 4 the weight changed from plus 0.4 at the end of two days to minus 14.0 per cent at the end of nine days. Work with deaminized hide showed that deamination raises the shrinkage temperature of hide considerably: also that vegetable tannage has very little effect on the amount of acid that will combine with either normal or deaminized collagen. From work done by others the author points out that the reduction of fixed tannin is proportional to the number of basic groups removed by deamination, giving evidence that the tannin is combined with basic collagen side chains. It is possible that strong acids displace the tannin from combination with these groups. The author discusses the data presented as compared to and correlated with literature on the same subject at some length, particularly emphasizing the importance of steric factors in actual tanning. The following conclusions are drawn. In vegetable tanning, tannin combines first with the basic groups of collagen and probably links them by hydrogen bonds to the peptide groups. It is the tannin attached to the arginine groups which raises the shrinkage temperature. Subsequent to this firm fixation, tannin is loosely bound, probably by single point hydrogen bonding to the peptide chains, this combination lowering the shrinkage temperature. These reactions are followed by a slow combination of tannin by multi-point hydrogen bonds to the peptide chains, probably replacing the short link of 4.5 Å between collagen chains by a collagen-tannin-collagen linkage. There is also a slow replacement of small tannin molecules by larger ones. It is possible that in all types of tannage, there is a general mechanism raising the shrinkage temperature in which the tanning material links a basic or acidic side chain to the peptide group. The total amount of tannin combined with the hide is largely determined by the volume available for it and the accessibility of the reactive groups. R. H. T.

Use of Sodium Silicofluoride During the Curing of Hides and Skins. C. C. Kritzinger. *J. Soc. Leather Trades' Chemists*, **37,** 193 (1953). The literature covering the use of fluorine compounds, particularly sodium silicofluoride, for the past fifty years is reviewed. The consensus of opinion now is that the fluorides and silicofluorides are excellent bacteriostatic if not bactericidal agents, that their use is outstandingly beneficial to the curing process and that they will not adversely affect the quality of the leather. Four different methods of use are described and their practicality and defects discussed. The most efficient method for hides and calfskins is that of dipping the stock for a period of ten minutes to forty-eight hours in a saturated or nearly saturated solution of sodium silicofluoride prior to curing. Sheep and goatskins cannot be treated in this manner due to excessive wetting. The stock may be cured by any recognized method after this treatment. An Italian patent in 1929 and a U. S. patent in 1932 covering the dipping method probably have retarded the investigation and use of this method. The second best method for hides and calfskins is to mix not less than 0.2 per cent sodium silicofluoride on the solution with brine, the stock being brined for at least 16 hours prior to further salting. Again this method is not applicable to sheepskins and goatskins. For proper results it is recommended that 2 lb. of the silicofluoride be mixed with every 10 gallons of brine when newly prepared and that

the solution be strengthened by adding one pound of the silicofluoride to every 10 gallons of brine after each batch dipped. The third method, in order of efficiency, is to mix 2 lb. of sodium silicofluoride with every 100 lb. of curing salt, taking care to secure maximum intimacy of mixture. This method can be used for all types of hides and skins. The least efficient method is to spray the flesh side of all hides and skins immediately after flaying with a 5 per cent mixture of sodium silicofluoride and water, using one gallon to every 5 hides or 20 skins. The tendency of such mixtures to settle can be counteracted by the addition of an anionic surface active agent. It should be pointed out that the advantages of one method over another are slight. The final selection may well be decided by the practical conditions under which curing is carried out. R. H. T.

Note on the Selection of a Substrate for Testing Enzyme Bates. W. G. Crewther and T. A. Pressley. *J. Soc. Leather Trades' Chemists*, **37**, 203 (1953). In the manufacture and use of bates it is desireable to have a reliable measure of the activity of the material in order to standardize the product. Test methods were studied by the International Commission for the Analysis of Artificial Bates and, although the Lohlein-Voolhard method involving the degradation of casein was considered satisfactory for pancreatic bates, it was less suitable for bacterial bates. Attempts to reach agreement on the adoption of a standard method were abandoned. The bating action of pancreatic extracts and the product of mold or bacteria growth is due to the breakdown of the *arrector pili* muscle attachment, the amino acid content of which may be unrelated to that of collagen. Some elastin and collagen degradation may also take place. In choosing a method for the standardization of bates it is highly desireable to select one which estimates the activity of all the enzymes responsible for bating. Thus gelatin, which contains no tyrosine but a considerable portion of arginine and lysine, it rapidly hydrolysed by trypsin, but is a poor substrate for chymotrypsin. Casein and denatured haemoglobin, both of which contain tyrosine, are better substrates for the estimation of chymotrypsin activity and are also hydrolysed readily by trypsin and other proteases. Since trypsin is relatively unstable between the pH values of 7 to 9 at room temperature, whereas chymotrypsin is relatively stable under these conditions, it is possible that many bate preparations contain considerable amounts of active chymotrypsin but rather low tryptic activity. Such preparations may have adequate bating properties but low activity when measured by the rate of digestion of gelatin. On the basis of the theoretical considerations, methods which depend upon the digestion of gelatin or hide powder may give misleading data and should be discarded. Although the methods using casein may not exactly parallel the bating activity of a series of enzyme preparations, the presence of all the amino acids in casein recommends it in preference to substrates which are deficient in particular amino acids. The use in parallel of two substrates would insure still more effective control. R. H. T.

A Comparative Study of the Effects of Various Acids and pH Values on Sole Leather Yields. S. G. Shuttleworth. *J. Soc. Leather Trades' Chemists*, **37**, 206 (1953). The object of these experiments was to compare the effect of lactic, formic and sulfuric acids at three levels of pH control. This involved setting up nine pilot plant runs, of two to three weeks each, involving approximately 180 to 250 sole leather squares 8″ x 10″ in area in each run. Adjustment of pH with the acid under test was confined to the first three deliming suspenders of the belly and butt liquors only, no acid being added to the remaining liquors, which were fed from the top butt handler at 100°BK. made from standard wattle extract of average pH value of 4.8. The total time of tannage was 15 days, including two Sundays in each series of 13 pits when no movement of hides took place. Commencing with 100°BK.

wattle extract, the liquors passed down through the four butt handler pitts, across to four belly handler pits, back to six butt suspenders, back to three butt deliming suspenders (acidified). The layout and analyses of the tan liquors, the average pH of liquors 24 hours after adjusting pH and yields of leather are given in tabular form. The results indicate that for the changes described, which involved the use of sulfuric acid, formic and lactic acids to adjust the pH values of the deliming suspenders to 3.0, 3.5 and 4.0, the average wet yield expressed on the limed weight does not vary greatly. The small variation suggests that the yield depends on the relation between pH value and salt content of the early tan liquors. If the pH is controlled at too low a value for the amount of salt present in the liquors poor penetration of the tan may cause a drop in yield, depending on the length of tannage and thickness of hide. This is evidently due to swelling of the fibers, which must be controlled to the correct amount to give good tan fixation combined with good penetration into the center of the hide. Sulfuric acid gave poor pH control on account of its low buffering power. It also caused sludge formation. Care should be exercised in its use.—R.H.T.

Fluorescence Microscopy in the Tannery. By F. Witke. *Oesterreichische Leder-Zeitung* **8,** 43 (1953). The principles of fluorescence microscopy are reviewed. Preparations, either unfixed or fixed with formol, that show little differentiation may be examined by secondary fluorescence. For this purpose the preparation is treated for a few seconds or minutes with very dilute solutions of certain dyes or colorless compounds that fluoresce strongly. These dyes have been called fluorochromes. A frozen section may be prepared, stained and examined in a few minutes. Berberine sulfate for example, stains connective tissue blue, the cell nucleus bright yellow, and fat carmine red. Acridineorange is an almost universal fluorochrome. The outer horny layer of a frozen section of a fresh beef hide is made to fluoresce orange red by acridineorange; the stratum germinativum, a cloudy orange; the collagen of the papillar layer and the stratum reticular bright blue; the center of the collagen fibers, bright yellow green; the fat glands, greenish yellow; the erector pili muscle, brownish green. The hair bulb has an orange red fluorescence which changes along the hair to greenish blue, then to pure blue at the tip. Elastin fibers could be detected in bated or tanned skins by the fluorescence microscope although they could not be seen by the usual methods with the light microscope. Fibroblasts have been thought to be digested or altered by bating because they can not be detected by methods using daylight microscopy; they can however, be detected with a fluorochrome. I.D.C.

Disinfection and Conservation in the Leather Industry, with Special Consideration of Hide Preservation. By W. Hausam. *Oesterreichische Leder-Zeitung* **8,** 77 (1953). A review lecture. Naphthalene-soda-salt is effective, but the soda separates easily. Fluorides, including silicofluorides, are not as satisfactory as they promised to be, and sometimes result in ribbed leather. There is the problem of toxicity of gelatin prepared from hides cured with salt, containing fluorides. Quaternary ammonium compounds are very irritating to nasal and mucous membranes. Zinc oxide is an effective preservative but ruins photographic gelatin. In soaks, bacterial action is stopped by 1:250 parts of phenol, 1:1000 of para-chlor-meta-cresol (Raschit K) or 1:2000 of mixed chlorinated phenols. The use of Raschit in various tannery operations is described. I.D.C.

Practical Experience with the Pretreatment and Paste Drying of Different Sorts of Leather. By Hanna Esser. *Oesterreichische Leder-Zeitung* **8,** 81 (1953). Many types of leather are suitable for paste drying. Some of these are chrome tanned box side

leather containing from 2.8 to 3.5 percent of chromic oxide (Cr_2O_3), combination tanned upper leather, chrome tanned patent leather, vegetable-syntan tanned sandal leather, combination or vegetable-syntan tanned bag and case leather, clothing leather, split leather, etc. Detailed directions are given for tanning and paste drying box side leather. For paste drying the sides should be split from 1/10 to 2/10 thicker than usual. Special fatliquors are required to produce plump, elastic leather rather than flat, tinny leather. Two phase fatliquors consist of a "deep" liquor or strongly sulfated oil which tends to penetrate deeply and a "surface" liquor or coarsely dispersed oil, approximately two-thirds sulfated, which tends to deposit on the leather surface. For box side leather from 1/2 to 2/3 of the oil should be deep liquor type. For combination tanned leather containing over 7 percent retan tannin, 3/4 of the liquor should be deep liquor. For chrome leather 1/3 more, and for combination tanned leather 1/2 more fatliquor should be used than for toggled leather. The surface liquor should be added only after the deep liquor has penetrated well. A modification of the method is to reserve 1/3 of the deep liquor and apply it with a sponge or brush to the thicker portions of the skin on the flesh side after the leather has been pasted to the plate. After fatliquoring the leather should be left from 6 to 8 hours then wrung under moderate pressure so that droplets of water will appear on the surface when the leather is pressed together. The requirements of pastes and mode of application are given in general terms. The drying equipment of Benno Schiloe Machine Works is briefly described. I.D.C.

The Importance of Cattle Breeds in Leather Production with Notes on Some Typical Hide Damages. By W. Hausam. *Oesterreichische Leder-Zeitung* **8,** 99 (1953). The relative number of cattle (in Germany) of lowland breeds is increasing at the expense of upland breeds, although tanners consider hides from the latter superior to those from the former. From the examination of hides of 600 cows, it was found that those from upland breeds averaged 0.85 millimeters thicker than those from lowland breeds. Hide weight, both absolute and weight relative to animal weight, was related to age of animal and passed through a minimum at 8 or 9 years of age. Follicular mange is increasing enormously not only in Germany, but apparently throughout the world. Lots have been found in which 85% of the hides were infected. I.D.C.

Silica Tannage. II. Diffusion of Silica Sols Into Gelatin Gels. P. Chambard and R. Lasserre. *Bull. Assoc. Franc. Chimistes Inds. Cuir* 13, 179-86 (1951). Iron-silica sols were prepared by pouring 50 ml. of a sodium silicate solution (10% SiO_2, SiO_2/Na_2O = 2.8) into 7.5 ml. of 10 *N* HCl, followed by a variable amount of $Fe_2(SO_4)_3$ solution (8.7% Fe) and H_2O to make 100 ml. A 10% gelatin gel was prepared and brought to pH = 1.8. Fifty g. portions of gel were placed in dishes, melted, and cooled. Fifty ml. of the SiO_2 sols were added and let stand 8 days. The sols contained 0, 16, 30, 51, and 70 parts Fe per 100 SiO_2, and one solution contained $Fe_2(SO_4)_3$ only. In all cases where SiO_2 was present, the gel contracted. The amount of contraction, judged from the volume of supernatant liquor, increased with ratio of Fe to SiO_2, but contraction was less when only Fe was present. A very thin, hard layer, insoluble in boiling H_2O, formed at the gel-liquor interface. After 8 days the gels were split into 3 layers and analyzed. The SiO_2 content of the surface layer declined from 18 to 5% with increasing Fe content of the liquor; that of the middle layer from 3.4 to 0.9%; and that of the bottom layer from 2.3 to 0.3%. The Fe content of the gels increased with Fe content of the liquor, and for each liquor the Fe content of the gels increased from the surface to the bottom layer. The ratios of Fe to SiO_2 in the spent liquors were about 75% of the corresponding ratios in the initial liquors. Melting points of the tanned gels (exclusive of the surface layer) increased from 24 to 36°C. with increasing Fe

concentration. The authors conclude that (1) silica has a true tanning action, because of the insolublization of the surface layer, (2) the state of aggregation of SiO_2 in these sols is too great to permit much penetration, (3) the Fe exerts a definite, though very weak, tanning action even at the low pH value of 1.8, (4) no Fe- SiO_2 complex is formed, but (5) the Fe modifies the tanning action of the SiO_2 by rendering the sol more stable and reducing the amount of SiO_2 that is fixed. *III. The Silica-collagen Combination. ibid.* 187-91.—Both SiO_2 tannage and ZrO_2 tannage take place in very acid solution, and both involve fixation of large amounts of highly polymerized acids. Tests were made to see whether fixation of SiO_2 involves the NH_2-group. Two series of tanning tests were made with SiO_2 sol (5% SiO_2) prepared by pouring a solution of sodium silicate ($SiO_2/Na_2O = 4$) into HCl, to give a sol of pH = 1.2. One series was done with dehaired, delimed, dehydrated, resoaked skin at its normal pH value of 5, and the other with the same material at pH = 1.2. In both series the system was neutralized stepwise to pH = 3.8-4.0 over 2 days. Each series comprised 5 tannages (1) straight SiO_2 tannage, (2) same, on deaminized pelt, (3) straight Cr tannage, (4) Cr tannage and SiO_2 retannage, and (5) SiO_2 tannage and Cr retannage. Analysis of resulting leathers showed: (1) results for non-acidified and acidified pelt were practically the same, (2) deamination reduced SiO_2 fixation from 51-56 to 29-32 g. per 100 collagen, (3) pretannage with Cr reduced SiO_2 fixation by about the same amount, with displacement of about half the Cr during the SiO_2 retannage, (4) pretannage with SiO_2 did not lower subsequent Cr fixation, and no SiO_2 was displaced, and (5) deaminization had practically no effect on Cr fixation. These results support the hypothesis that SiO_2 is bound, in part at least, by NH_2-groups, and definitely not by COOH-groups. The lower SiO_2 fixation by Cr-tanned pelt may be due to the inability of the pelt to swell, thus affecting adversely the penetration of SiO_2, although binding of Cr to NH_2-groups may also be a factor. *IV. ibid.* 192-6. Tanning tests were made on bated calfskin, using SiO_2 sols prepared at pH = 3.2 by methods derived from the work of Willstättar and Kraut (*C. A.* 25, 4811). A solution of Na_2SiO_3 was added rapidly to a mixture of HCl and CH_3COOH at 10°C. The resulting sol (2.5% SiO_2) was stable for at least 5 days. The pelt was pickled with NaCl and either HCl or HCl + CH_3COOH. Tannage was done either with 7.5% SiO_2 on the bated weight, in 300% float, for 48 hours, or with 10% SiO_2 applied in 2 successive 24-hour treatments with 5% SiO_2 in 200% float. In both cases the final solution was neutralized to pH = 3.9 during 24 hours (total time 72 hours); then the skin was rinsed, neutralized to pH = 4.5, rinsed again, and air dried. In tests with 7.5% SiO_2 the leather contained about 20% SiO_2, and shrank at 73-75°C. With 10% SiO_2, the leather contained about 25% SiO_2 and shrank at 78°C. The latter leather was well tanned and supple, with a smooth, non-brittle grain. In tests with deaminized skin, fixation of SiO_2 was not lowered, and the shrink temperature was 81-83°C. H. B. M.

Sulfochlorinated Paraffins and Their Use in the Tannery. F. Grall. *Bull. Assoc. Franc. Chimistes. Inds. Cuir* 14, 25-34 (1953); cf C.A. 43, 4038*f*. An address. H. B. M.

Blotches on Leather and Skins and the Alteration of Tannin Brought About By Yellow Mold (Paecilomyces Variota). Henri Martin and Paul Carie. *Bull. Assoc. Franc. Chimistes Inds. Cuir.* 13, 199-226 (1951). Light colored blotches on vegetable tanned leather appeared periodically when the hides were taken from chestnut handler liquors. This tannery used oak bark liquor in the suspenders, followed by 6 chestnut and 2 quebracho liquors. The trouble was traced to deposition of ellagic acid, produced by the action of the mold on the chestnut tannin; however, the source of contamination was the preceding oak bark liquors. The damage occurred only when the mold conidia came in contact with the grain surface. Vegetative mold growth on the surface of the liquor did not produce these

blotches, which are quite different from mere deposition of sludge (bloom). Sodium pentachlorophenate proved effective as a preventative. Tests showed that this mold destroyed about 11% of the tannin in a 4% chestnut liquor (pH = 4.0) in 12 days, also 11% of the non-tannin and 21% of the initial sugars. For oak gall tannin, about 40% of the tannin was destroyed, but non-tannin and sugars increased by 70 and 136% respectively. For wattle, tannin decreased by 35%, non-tannin by 50%, and sugars by 89%. pH values of the liquors declined by not more than 0.2, and no volatile acids were produced. Quebracho was relatively immune to attack by this mold. The action was most marked at pH values from 4.2 to 5.2. H. B. M.

Disk Chromatography and Its Application to Qualitative Tannin Analysis. A. Jamet. *Bull. Assoc. Franc. Chimistes Inds. Cuir.* 14, 19-23 (1952). An alumina disc is formed by allowing 3 ml. of special Al_2O_3 powder to flow from a wide orifice pipet onto a glass plate, forming a cone, and pressing to form a disk with another plate pierced by a 5 mm. hole. The disk should be about 8 cm. in diameter and 1 mm. thick. The solvent is *N*-butanol saturated with CH_3COOH and water (40 ml. butanol shaken with 10 ml. pure CH_3COOH and 50 ml. water, and the aqueous phase discarded). Tannin solutions (20% tannin) are diluted with an equal volume of ethanol or methanol and centrifuged. The solvent (0.4 ml.) is first added from a fine-tipped buret, dropwise, through the hole in the upper glass plate. After 5 minutes, 6 or 7 drops of the alcohol-tannin solution are added, and 5 minutes later another 0.4 ml. of solvent. Let stand 15 minutes. Lift off the upper plate without disturbing the disk, and examine in ordinary light and with ultraviolet light (Wood's lamp). The general pattern is a dark, central spot, surrounded by a series of rings. In ordinary light, chestnut gives a beige spot, next a colorless ring, finally a red ring at the limit of the chromatogram. Quebracho (sulfited) gives a reddish beige spot, from which rays of the same color protrude, and a yellow ring, partly traversed by the rays. Sumac gives a bright yellow central disk. Tannic acid, in alcohol, gives no color at all. In mixtures of chestnut and quebracho the yellow ring increases and the red ring decreases in intensity very noticeable as the proportion of quebracho is increased from 10 to 50%. The examination in ultraviolet light is more revealing. While exact verbal description is impossible, the general appearance is as follows: *Chestnut*—a very fluorescent violet ring surrounding the central spot. *Oak*—a very clear strongly fluorescent, sky-blue ring. *Myrobalans*—a maroon central spot with greenish luminescence, surrounded by a fluorescent violet ring and a yellowish border at the limit of the chromatogram. *Sulfited quebracho*—a maroon central spot, little fluorescent, surrounded by a very fluorescent yellow ring and a broad, very fluorescent sky-blue border. *Wattle*—a violet central spot, surrounded by a sky-blue, fluorescent ring, bordered in yellow; a second blue ring, in the shape of a festoom, limits the chromatogram. *Tannic acid* (in alcohol)—The central spot shows a bright reddish violet fluorescent and is surrounded by a narrow, indented, yellowish white ring. *Sumac leaves*—The central spot is very dark, but luminescent, surrounded by a bright golden yellow ring. After 24 hours a second yellow ring forms, separated from the first by a gray zone. *Valonia*—a greenish maroon central spot, surrounded by a pale violet, fluorescent ring. The chromatograms should be examined on the day of preparation, as the fluorescence loses its intensity after 2 days. H. B. M.

Quantitative Tannin Analysis. Filtration of Detannized Solutions Through a Porous Glass Filter. H. Martin and E. Grosjean. *Bull. Assoc. Franc. Chimistes Inds. Cuir* 14, 15-18 (1952). See Chambard and Jamet, *C.A.* 43, 8182*h*. The advantages of this procedure are elimination of errors caused by soluble matter in filter paper and kaolin,

considerable saving of time, and greater assurance of getting an optically clear filtrate. *Procedure:* Cut a disk of filter paper to fit exactly in the bottom of a Pyrex No. 172.211 filter crucible, No. 1 porosity, 40 mm. diameter and 50 mm. high. Moisten the paper with a few ml. of non-tannin solution, and apply moderate vacuum. Transfer the slurry of tanned hide powder and detannized solution to the crucible, a little at a time, to build up a uniform filter cake. Return the first portions of the filtrate to the crucible several times, until it comes through clear. Transfer the rest of the slurry to the crucible, in installments, never allowing the filter mat to suck dry. If the filtrate is not optically clear, pass it through the filter a second time. Be sure that the filter mat of tanned hide powder fills the bottom of the crucible completely without leaving an annular void around the edge. Results are said to agree with those obtained by the usual filter paper-kaolin procedure, when the paper is pre-washed. No data are given. The method is said to apply equally well to non-tannin determinations by Chambard and Jamet's direct method (using dry, prechromed hide powder) and by the official method, although it is not clear how the amount of hide powder ordinarily used could be handled in a filter crucible of the size specified. The potential amount of soluble matter in a 4-cm. disk of filter paper is negligible. H. B. M.

Amphoteric Tanning Materials. By G. Mauthe and K. Faber. *Das Leder* **4,** 49 (1953). Vegetable tannins are organic substances with phenolic and therefore weak acid character. Their tanning intensity depends greatly on the acidity of the tan liquor. Increasing the acidity of the liquor increases the affinity for hide substance, i.e., the astringency of the tan liquor, decreases the rate of penetration and results in an increase in the amount of tannin taken up. These effects are caused by a change in degree of hide swelling, an increase in particle size of the tannin and a change in the behavior of the weak acid tannin groups to animal hide. The weakly acid phenolic hydroxyls, which are the tanning active groups, are scarcely dissociated in the acid range; they tan because they are able to form proton bridges. At high pH values the phenolic hydroxyls become acidic or anionic but then they lose most of their tanning ability. Synthetic auxiliary syntans, the purest type of which is the condensation product of naphthalene sulfo acid with formaldehyde, have no tanning active groups similar to those of vegetable tannins. They form salt-type combinations with proteins. Exchange syntans have phenolic hydroxyls and only a few sulfo groups to make the molecule soluble in water. Like vegetable tannins, they combine with protein by proton bonding. Vegetable tannins and exchange syntans differ from auxiliary syntans but all are anion active so that anion activity is not a tanning characteristic. Chrome tanning differs from the above in that increase in acidity lowers astringency. Chrome tanning is started in strongly acid solutions, then near the end of the process the tanning intensity is increased by neutralization. The chrome probably is bound to hide by auxiliary valence, but to polar groups such as carboxyl, rather than to peptide groups. Amphoteric, phenol-aldehyde resins were made that were basic and soluble in hydrochloric acid but they did not tan. Meister, in 1943-4, made a phenolic tannin whose solubility in water depended on attaching acid to an amine group. This syntan made good leather by a process involving neutralization just as in chrome tanning. Normally tanned leather, when retanned with this new cationic tannin gave leather of hitherto unknown fullness. Later numerous other syntans were synthesized. Gerbstoff R52 contains 1.88% nitrogen, 6.68% chlorine, 23.2% hydroxyl and has a molecular weight of 663. On analysis by the filter method, as the concentration of the solution (grams tannin per liter) increased, the purity (tannin over soluble solids) first increased to a maximum between 6 and 20 grams tannin per liter, then decreased slightly. Vegetable tannins show only a decrease. With increasing acidity the tan and combining values of R52 of SR11, a similar but less astringent syntan, decrease whereas these values increase for quebracho. Practical tanning experiments with R52 showed that (1) a pickled hide is penetrated faster

than one that is not pickled, (2) with unpickled hide the liquor becomes more basic because of takeup of acid by the hide, and (3) liquor exhaustion is greater without pickling than with pickling. The tanning process with R52 resembles chrome tanning but the leather tanned with amphoteric tannins of this type resembles bark tanned leather in fullness, feel, surface appearance, increased area and amount of combined tannin. The syntan might be represented as a long chain with an OH (hydroxyl) group at one end and an NH_2 (amine) group at the other end. In acid solution the OH group is not, but the NH_2 is strongly ionized and the syntan is cationic. As neutralization is approached the OH and NH_2 groups combine by salt links or hydrogen bonds to form quite long chains and the molecule loses its cationic character. The behavior can be explained completely by the fact that the precipitation point is being reached. Perhaps the increase in astringency of vegetable tannins on acidification is only the result of an approach to the precipitation point. Further practical evaluation of this class of tannins is needed. For example, chrome leather can be retanned with no change in dyeing properties and vegetable tanned leather can be retanned with greatly improved fullness. I. D. C.

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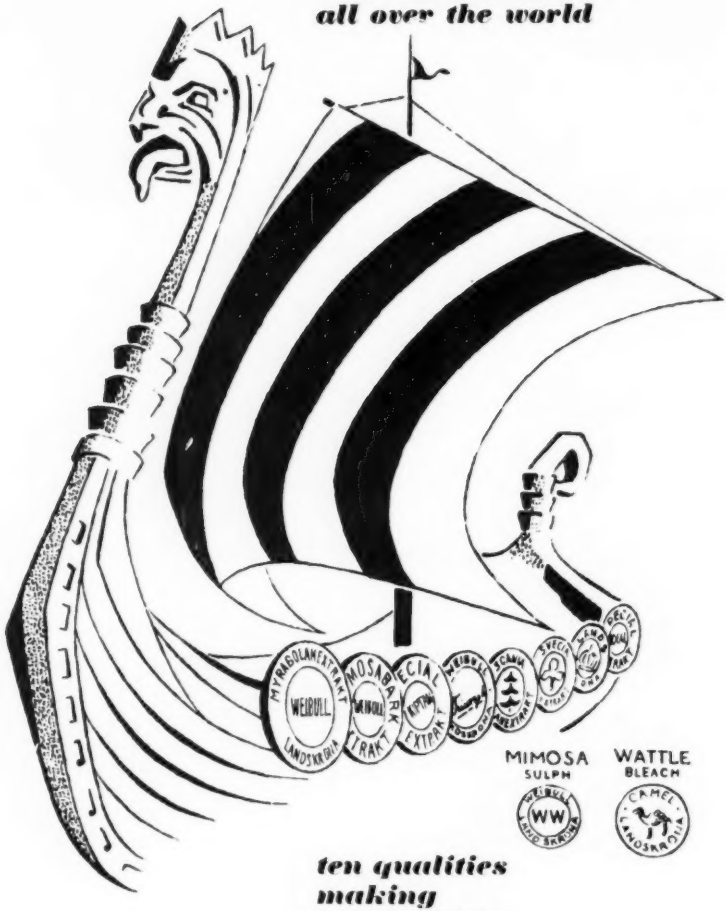

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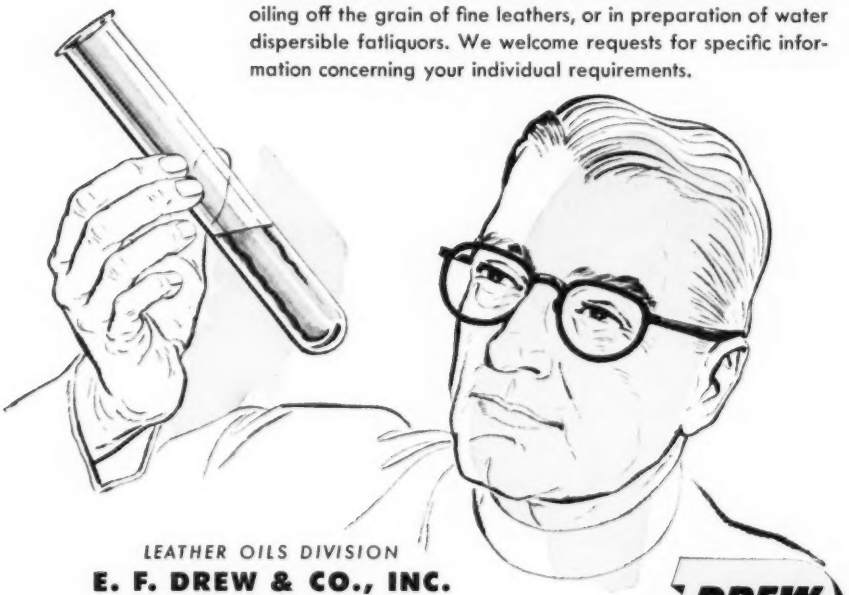

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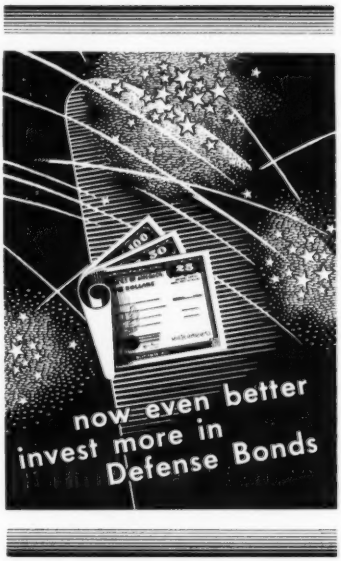

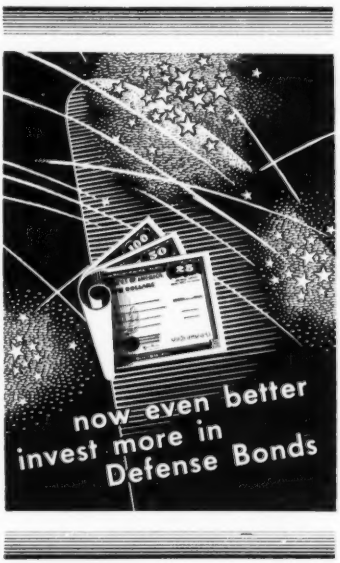

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INSOLUBLES	.65	.6	.7
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